Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Electronic Supporting Information

Practical Synthesis and Biological Screening of Sulfonyl hydrazides

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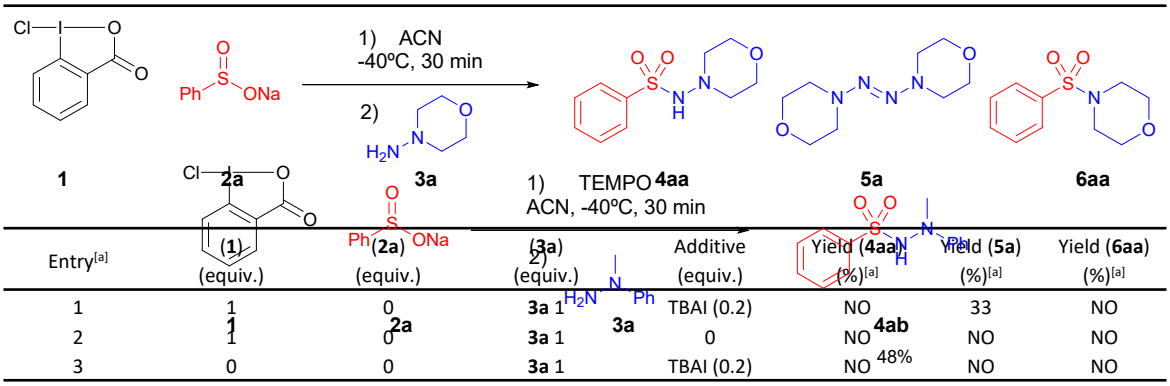
1. Complementary experiments

1.1. Control experiments

A round-bottom flask charged with chlorobenziodoxolone (**1**) (154.1 mg, 0.55 mmol) and TBAI (40.8 mg, 0.11 mmol). To the solids, 1,5 mL of solvent was added, and the reaction was stirred at - 40 °C for 30 min. The hydrazine, N,N-methylphenyl hydrazine (**3b**) (35 µL, 0.36 mmol) was then added, and the reaction stirred at -40 °C for 1 h. When completed, the reaction was allowed to warm up to room temperature, washed with a saturated sodium hydrogencarbonate solution, and the resulting aqueous phase was extracted with ethyl acetate. The organic phase was dried with sodium sulfate, filtrated, and concentrated under vacuum. The crude was purified using flash chromatography with ethyl acetate/hexane.

A round-bottom flask charged with chlorobenziodoxolone (**1**) (154.1 mg, 0.55 mmol), sodium phenylsulfinate (**2a**) (85.7 mg, 0.55 mmol), TBAI (40.8 mg, 0.11 mmol) and TEMPO (94.5 mg, 0.55

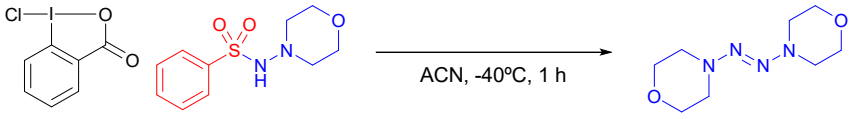


Entry[a]	(1) (equiv.)	(2a) (equiv.)	(3a) (equiv.)	Additive (equiv.)	Yield (4aa) (%)[a]	Yield (5a) (%)[a]	Yield (6aa) (%)[a]
1	1	0	3a 1	TBAI (0.2)	NO	33	NO
2	1	0	3a 1	0	NO	NO	NO
3	0	0	3a 1	TBAI (0.2)	NO	NO	NO

[a] Isolated yields; NO – Not Observed.

mmol). To the solids, 1,5 mL of solvent was added, and the reaction was stirred at - 40 °C for 30 min. The hydrazine, N,N-methylphenyl hydrazine (**3b**) (35 µL, 0.36 mmol) was then added, and the reaction stirred at -40 °C for 1 h. When completed, the reaction was allowed to warm up to room temperature, washed with a saturated sodium hydrogencarbonate solution, and the resulting aqueous phase was extracted with ethyl acetate. The organic phase was dried with sodium sulfate, filtrated, and concentrated under vacuum. The crude was purified using flash

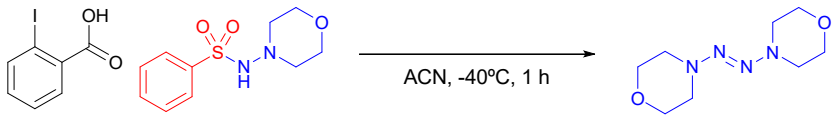
chromatography with ethyl acetate/hexane. The sulfonyl hydrazine N-morpholino-benzenesulfonamide (**4aa**) was isolated in 48% yield.



1 **4aa** **5a**

Not Observed

A round-bottom flask charged with chlorobenziodoxolone (**1**) (154.1 mg, 0.55 mmol) and N-morpholino-benzenesulfonamide (**4aa**) (128.7 mg, 0.55 mmol). The reagents were dissolved in 1,5 mL of solvent, and the reaction was stirred at - 40 °C for 1 h. When completed, the reaction was allowed to warm up to room temperature, washed with a saturated sodium hydrogencarbonate solution, and the resulting aqueous phase was extracted with ethyl acetate. The organic phase was dried with sodium sulfate, filtrated, and concentrated under vacuum. No compound **5** was obtained.



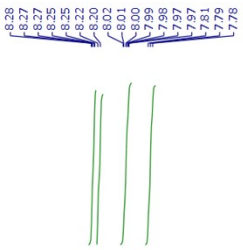
4aa **5a**

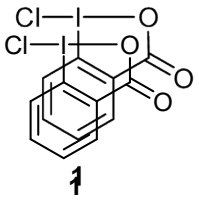
Not Observed

A round-bottom flask charged with 2-iodobenzoic acid (150.1 mg, 0.60 mmol) and N-morpholino-benzenesulfonamide (**4aa**) (146.5 mg, 0.60 mmol). The reagents were dissolved in 1 mL of solvent, and the reaction was stirred at - 40 °C for 1 h. When completed, the reaction was allowed to warm up to room temperature, washed with a saturated sodium hydrogencarbonate solution, and the resulting aqueous phase was extracted with ethyl acetate. The organic phase was dried with sodium sulfate, filtrated, and concentrated under vacuum. No compound **5** was obtained.

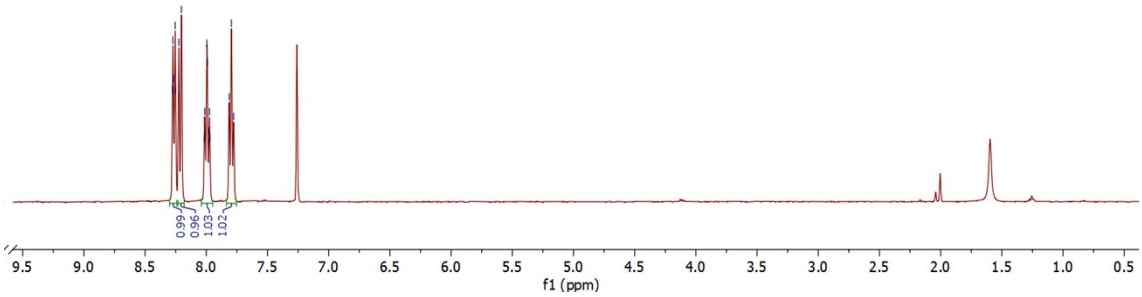
2. ^{1}H and ^{13}C spectra

2.1. ^{1}H NMR ($CDCl_3$, 400 MHz) of 1-chloro-1,2-benziodoxol-3-(1*H*)-one (1)

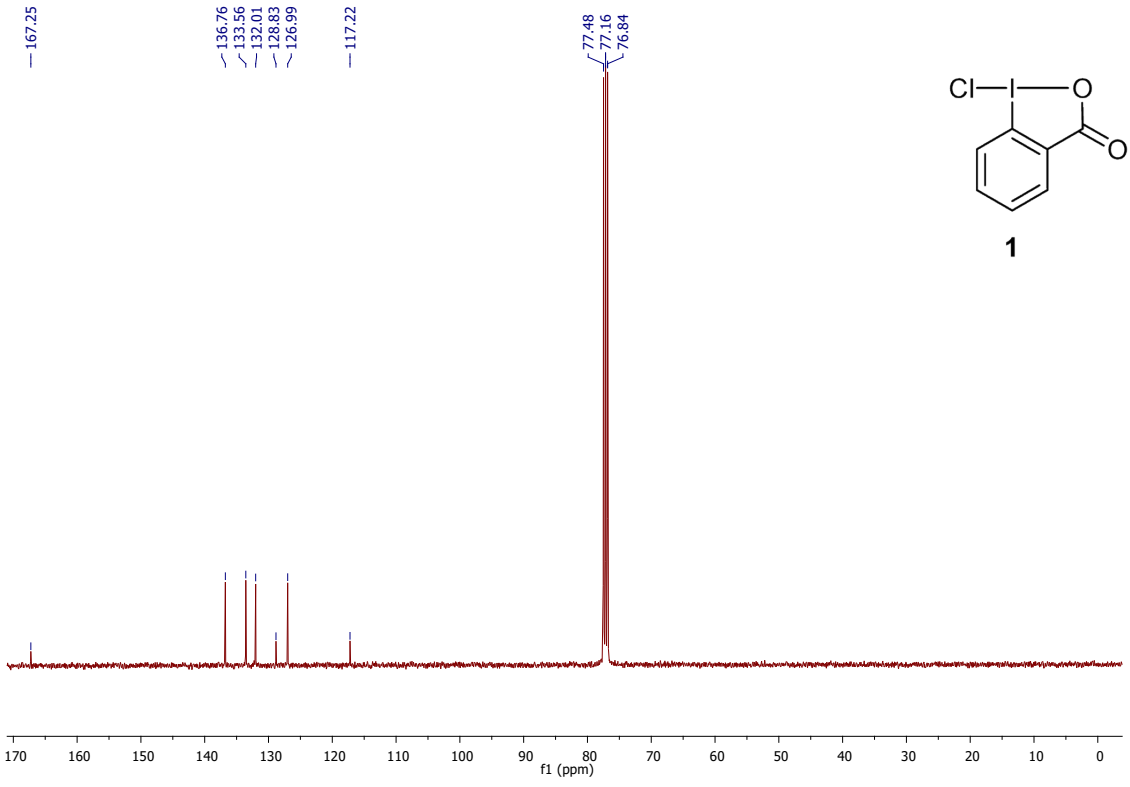




1

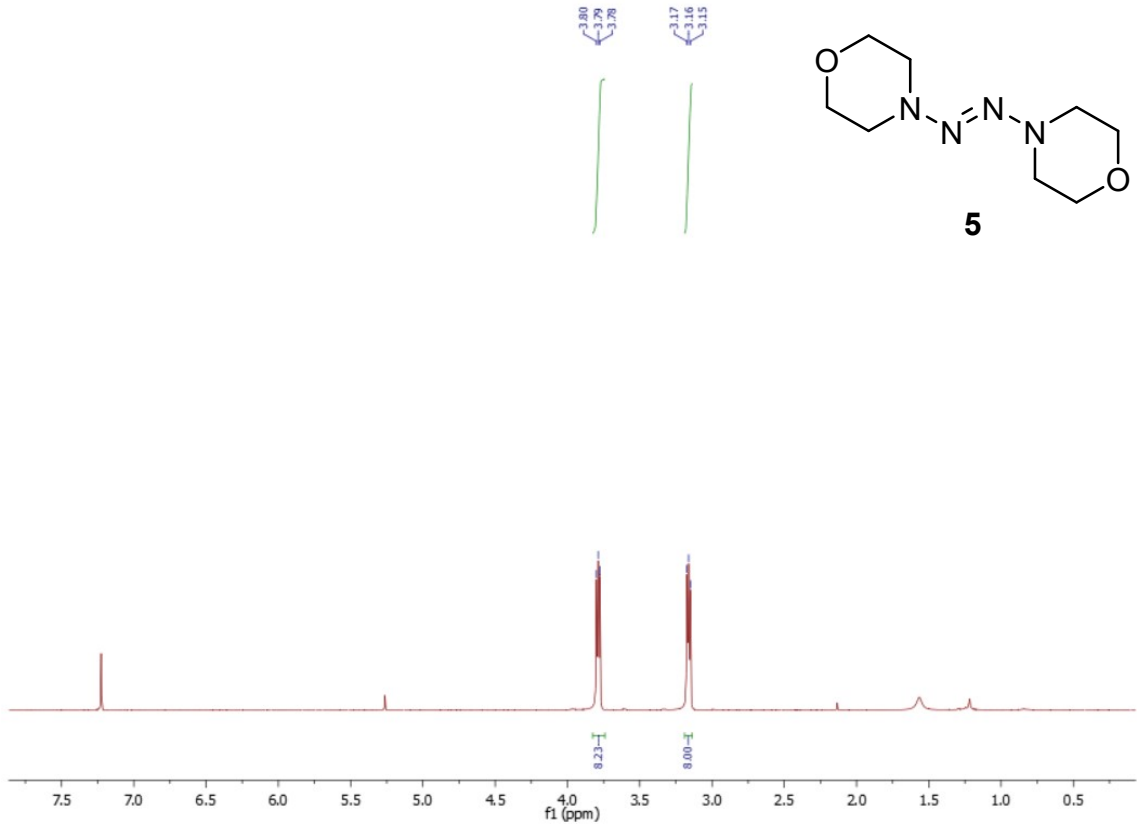


2.2. ^{13}C NMR ($CDCl_3$, 400 MHz) of 1-chloro-1,2-benziodoxol-3-(1*H*)-one (1)



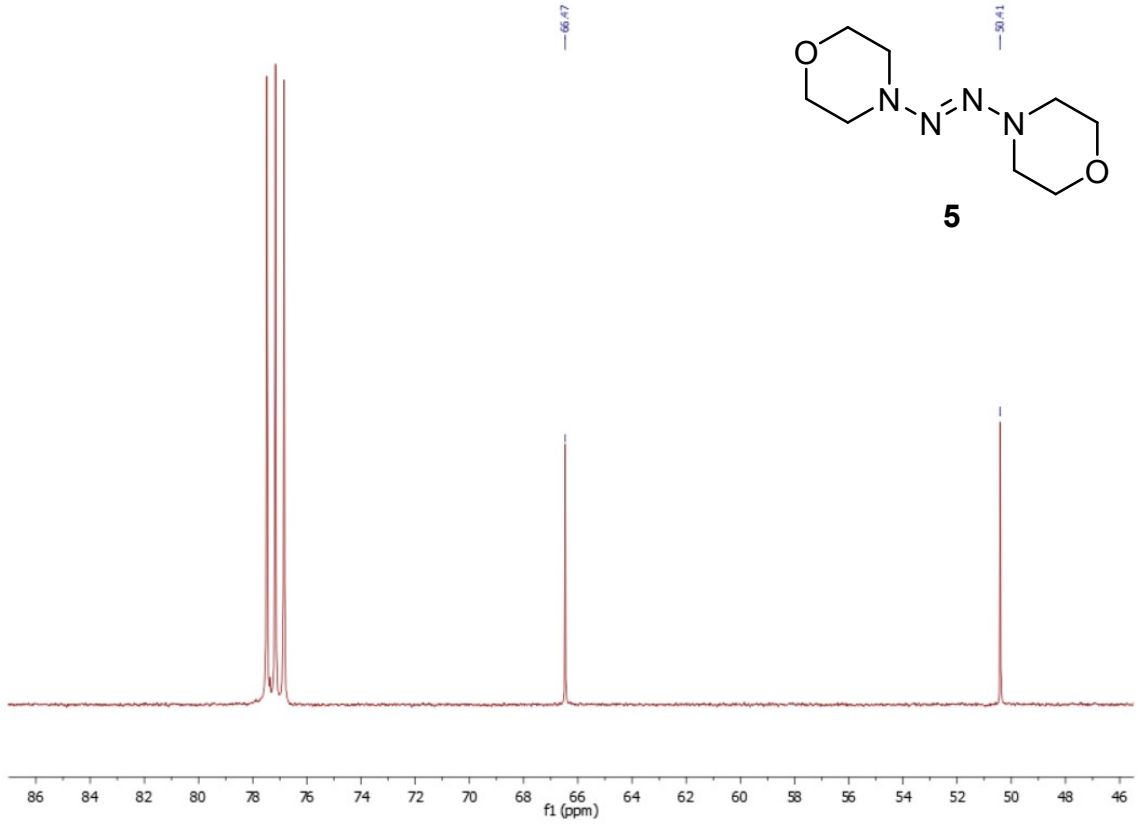
1

2.3. ^{1}H NMR ($CDCl_3$, 400 MHz) of 1,2-dimorpholinodiazene (5)



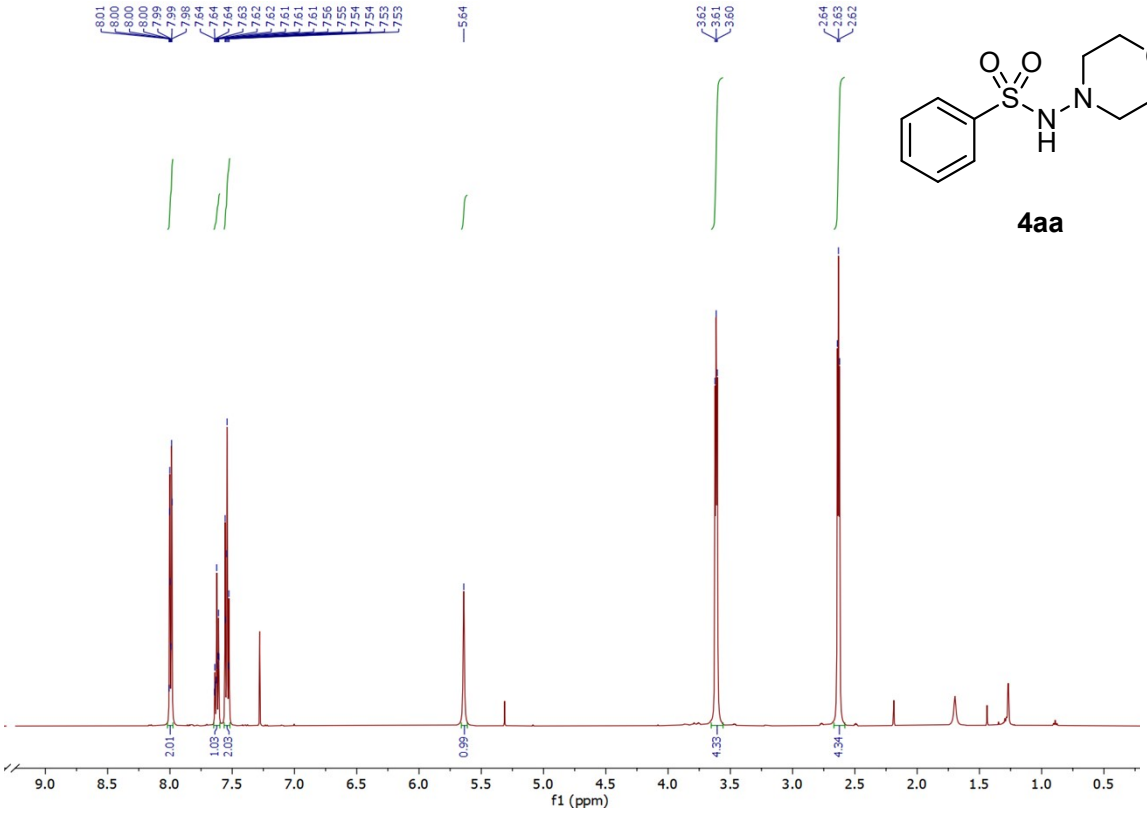
5

2.4. ^{13}C NMR ($CDCl_3$, 400 MHz) of 1,2-dimorpholinodiazene (5)



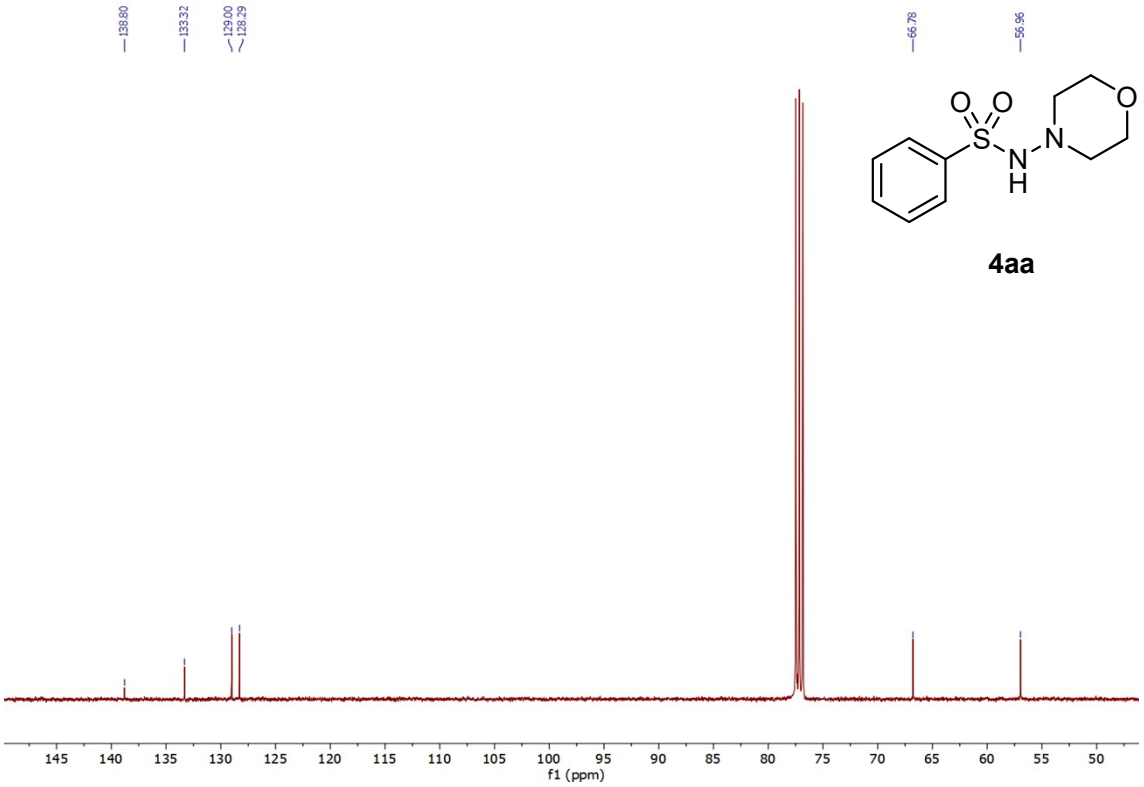
5

2.5. ^{1}H NMR ($CDCl_3$, 400 MHz) of N-morpholinobenzenesulfonamide (4aa)



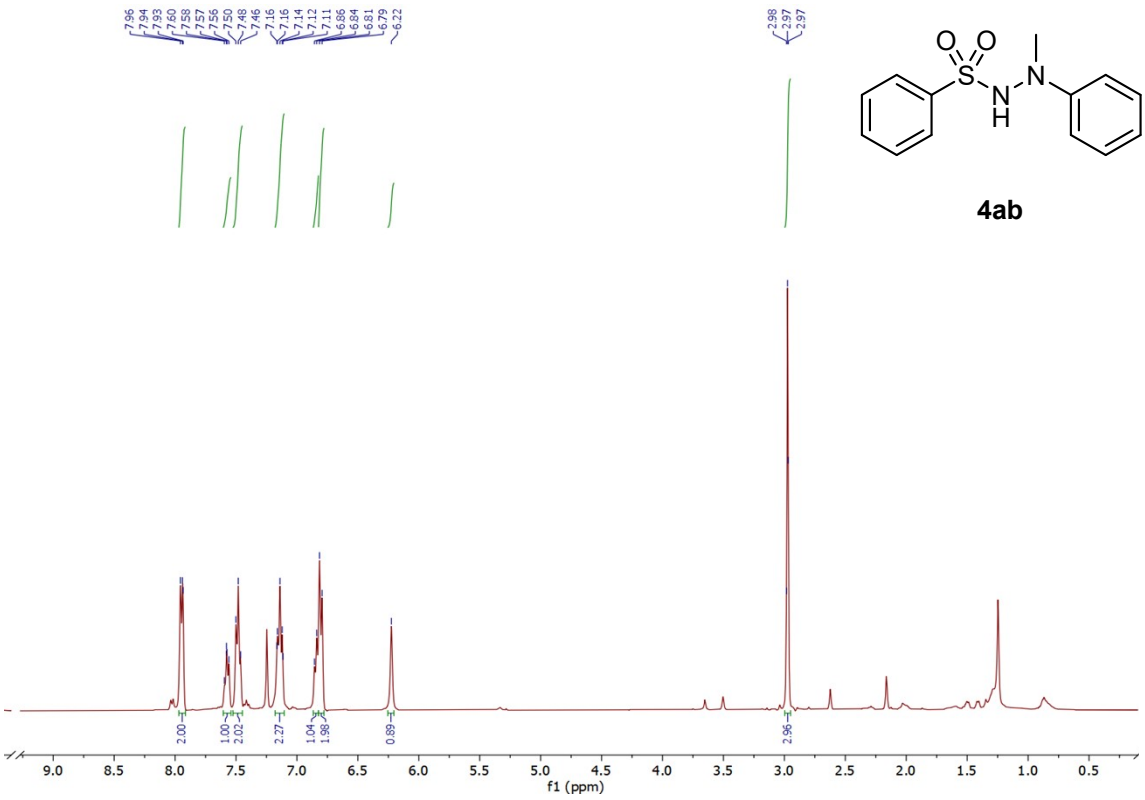
4aa

2.6. ^{13}C NMR ($CDCl_3$, 400 MHz) of N-morpholinobenzenesulfonamide (4aa)



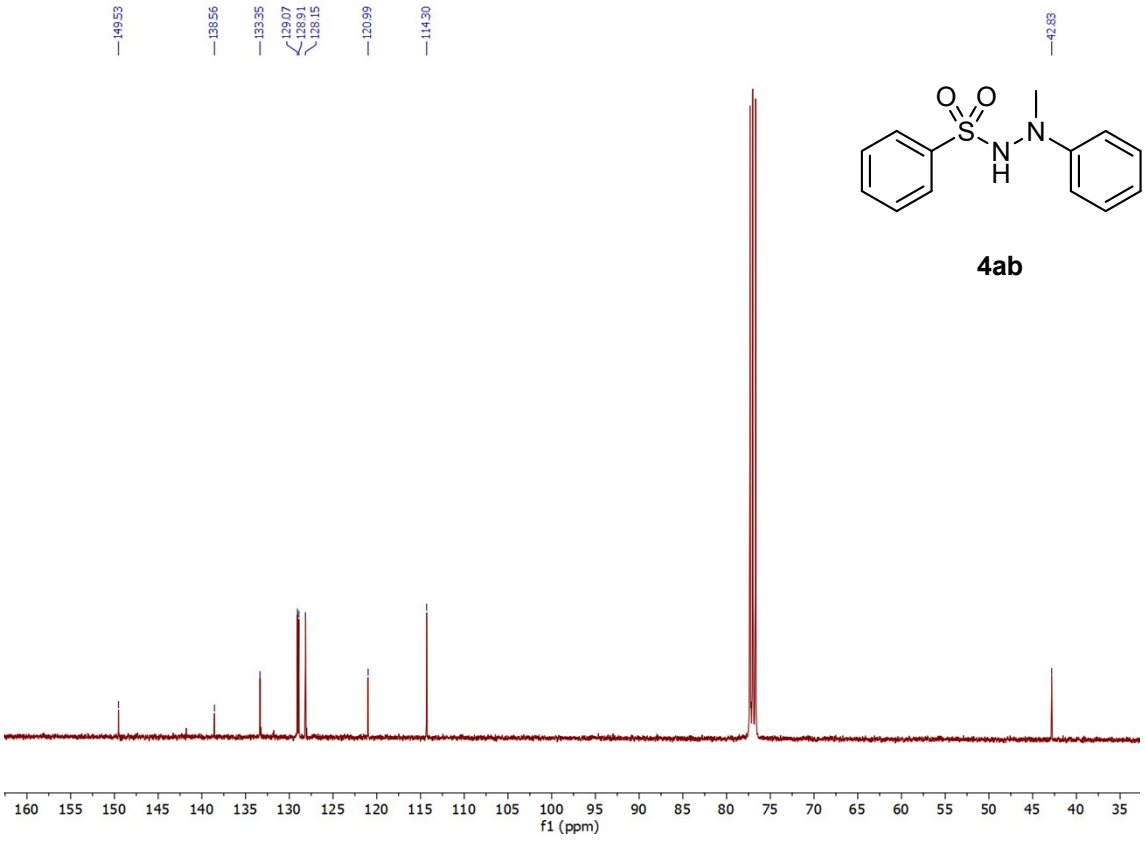
4aa

2.7. ^{1}H NMR ($CDCl_3$, 400 MHz) of N'-methyl-N'-phenylbenzenesulfonohydrazide (4ab)



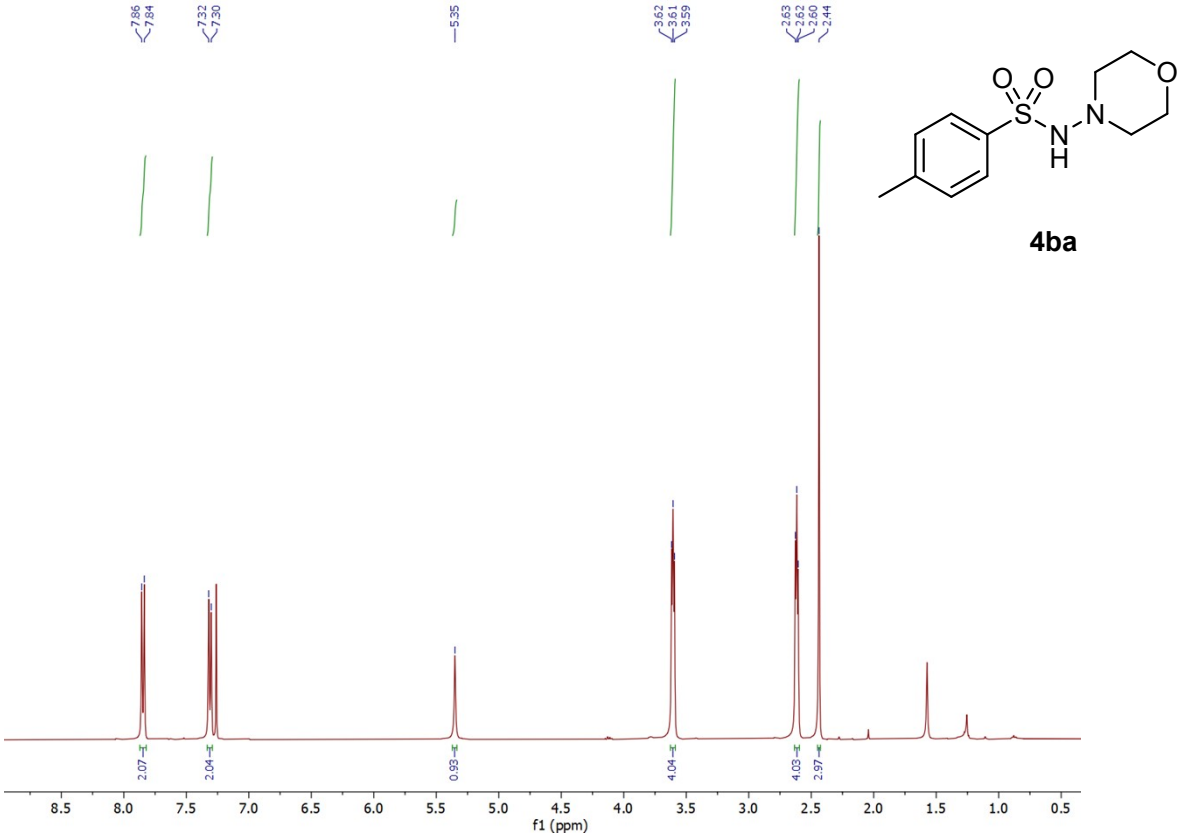
4ab

2.8. ^{13}C NMR ($CDCl_3$, 400 MHz) of N'-methyl-N'-phenylbenzenesulfonohydrazide (4ab)



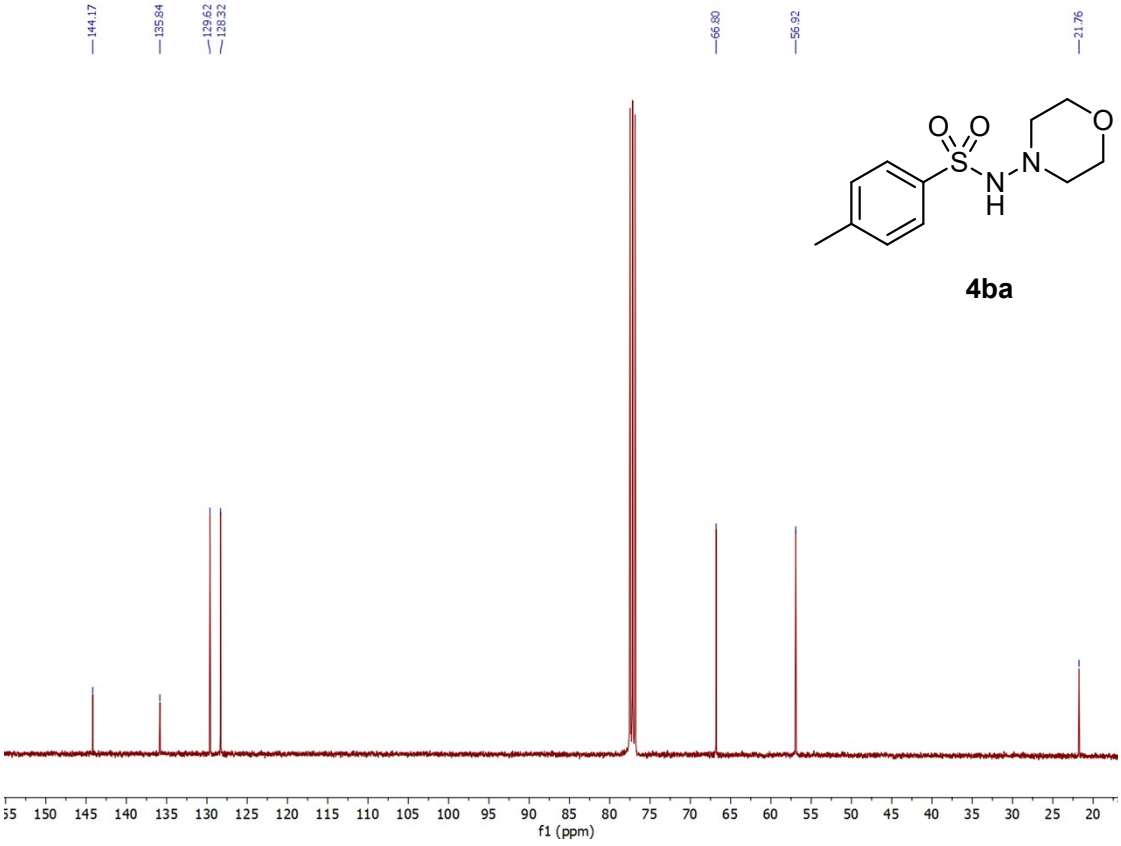
4ab

2.9. ^{1}H NMR ($CDCl_3$, 400 MHz) of 4-methyl-N-morpholinobenzenesulfonamide (4ba)



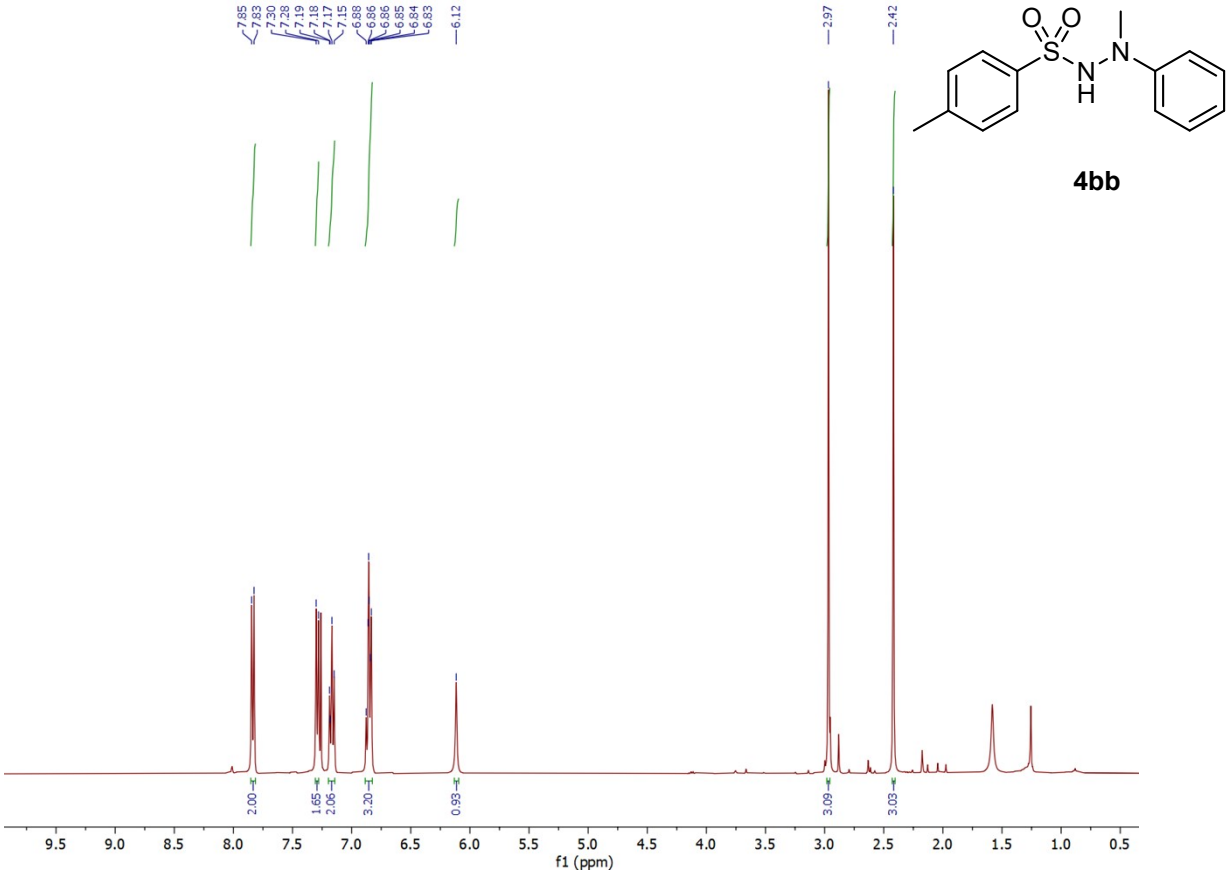
4ba

2.10. ^{13}C NMR ($CDCl_3$, 400 MHz) of 4-methyl-N-morpholinobenzenesulfonamide (4ba)



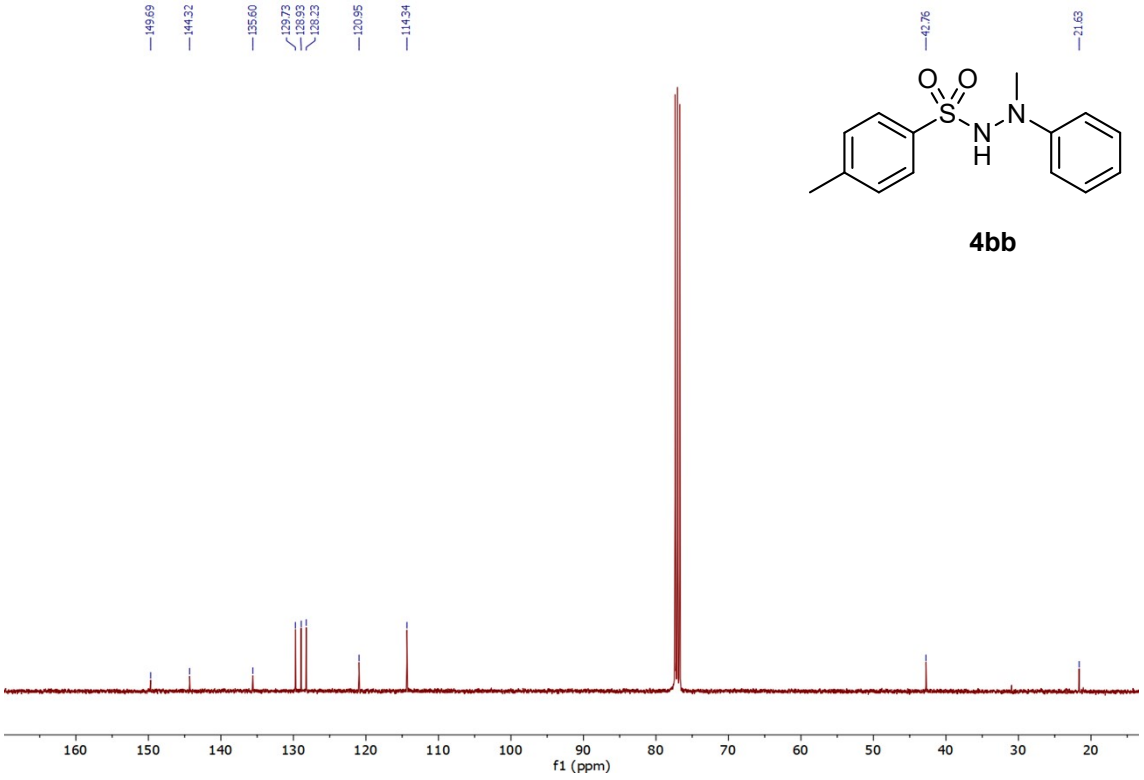
4ba

2.11. ^{1}H NMR ($CDCl_3$, 400 MHz) of N-Methyl-N-phenyl-N'-p-toluolsulfonyl-hydrazin (4bb)



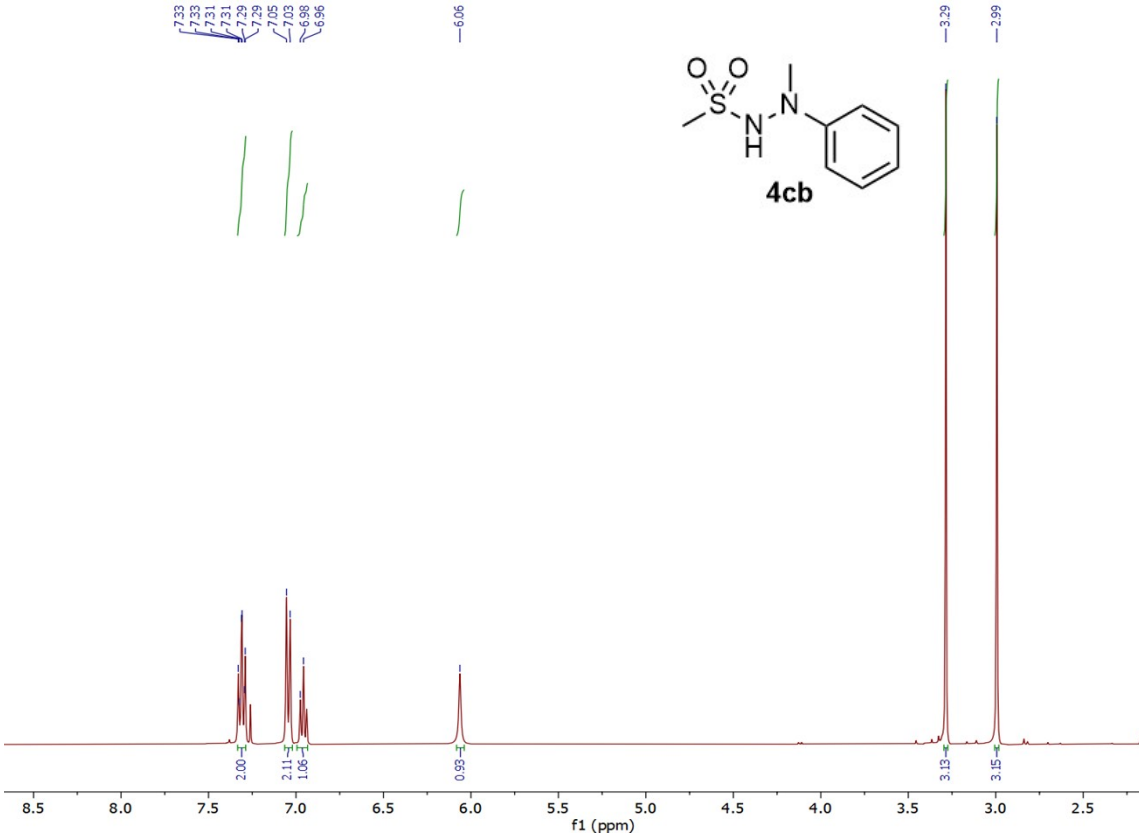
4bb

2.12. ^{13}C NMR ($CDCl_3$, 400 MHz) of N-Methyl-N-phenyl-N'-p-toluolsulfonyl-hydrazin (4bb)

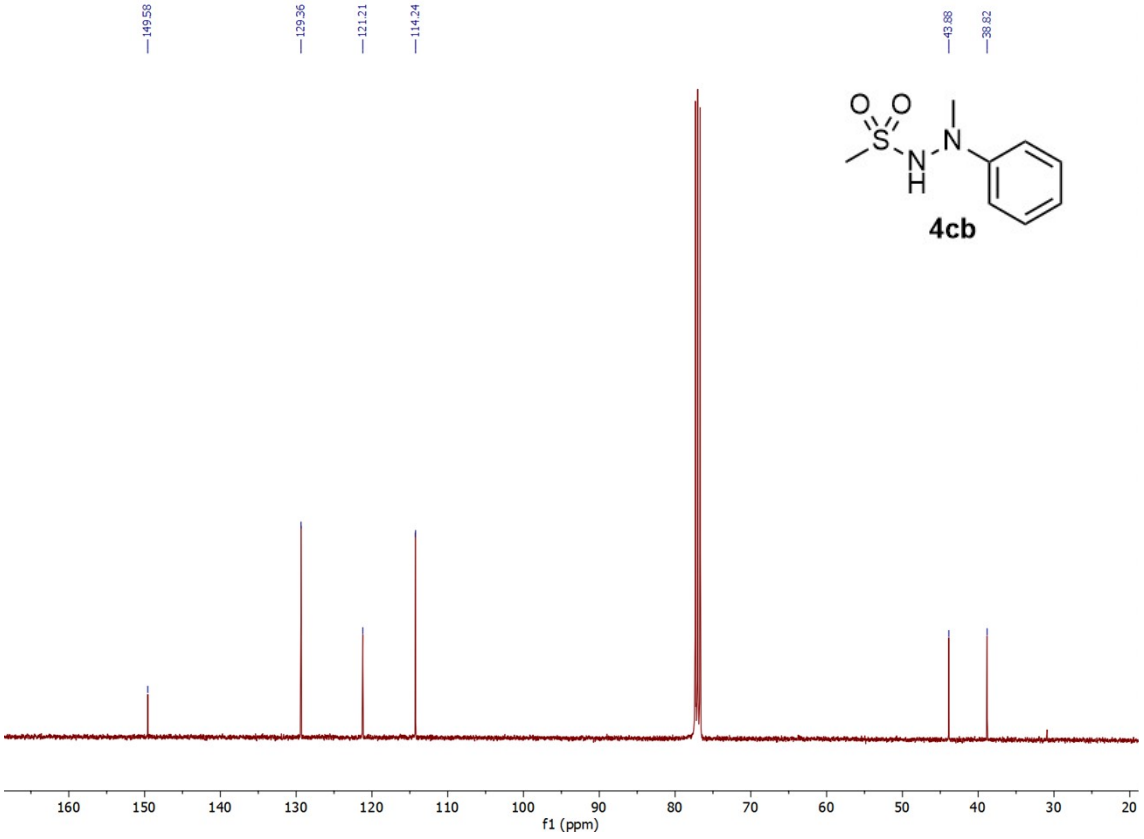


4bb

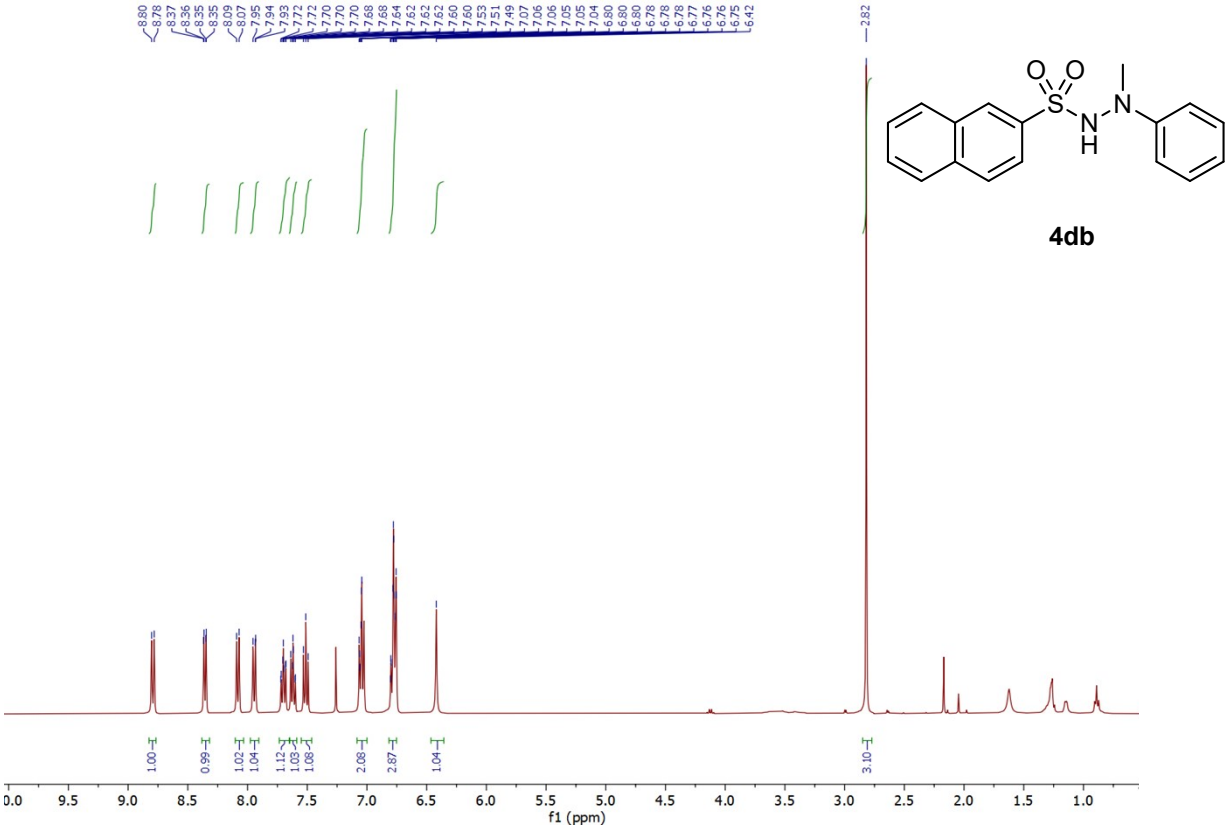
2.13. ^{1}H NMR ($CDCl_3$, 400 MHz) of N'-methyl-N'-phenylmethanesulfonohydrazide (4cb)



2.14. ^{13}C NMR ($CDCl_3$, 400 MHz) of N'-methyl-N' phenylmethanesulfonohydrazide (4cb)

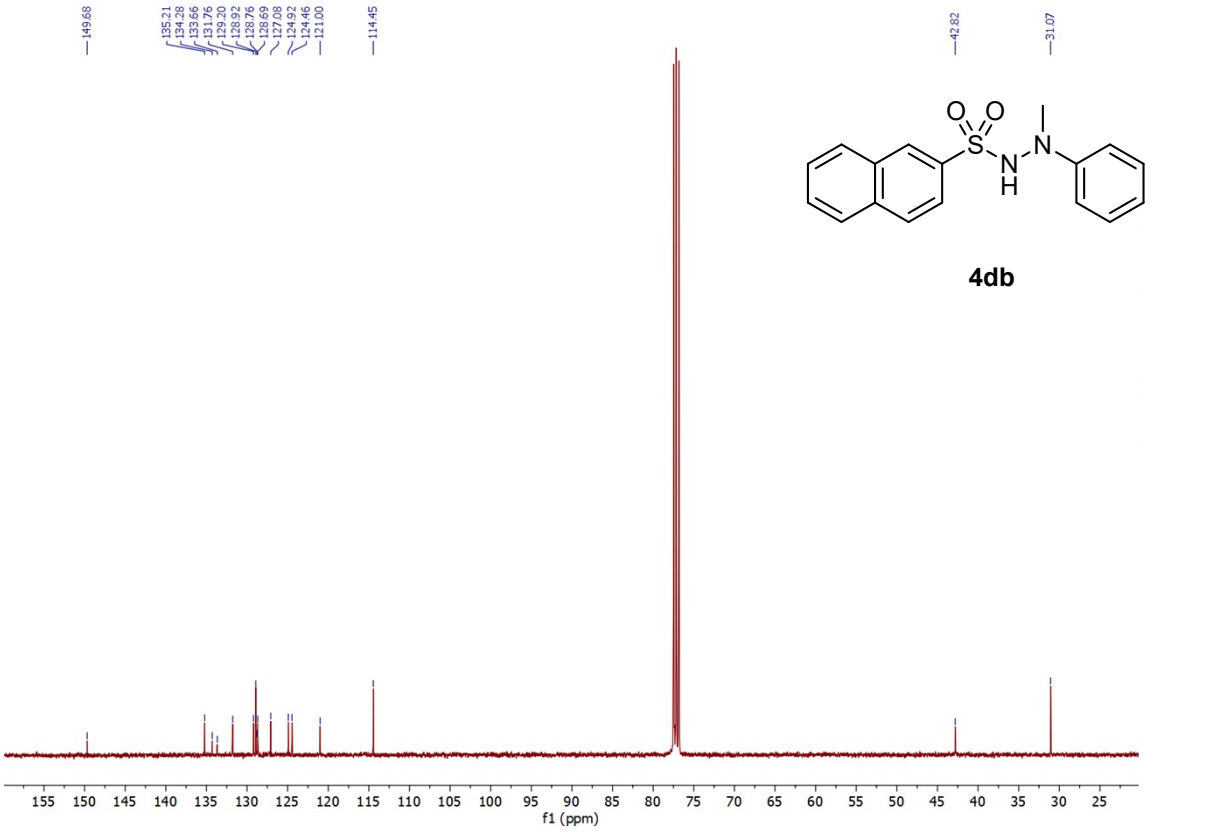


2.15. ^{1}H NMR ($CDCl_3$, 400 MHz) of N'-methyl-N'-phenylnaphthalene-2-sulfonohydrazide (4db)



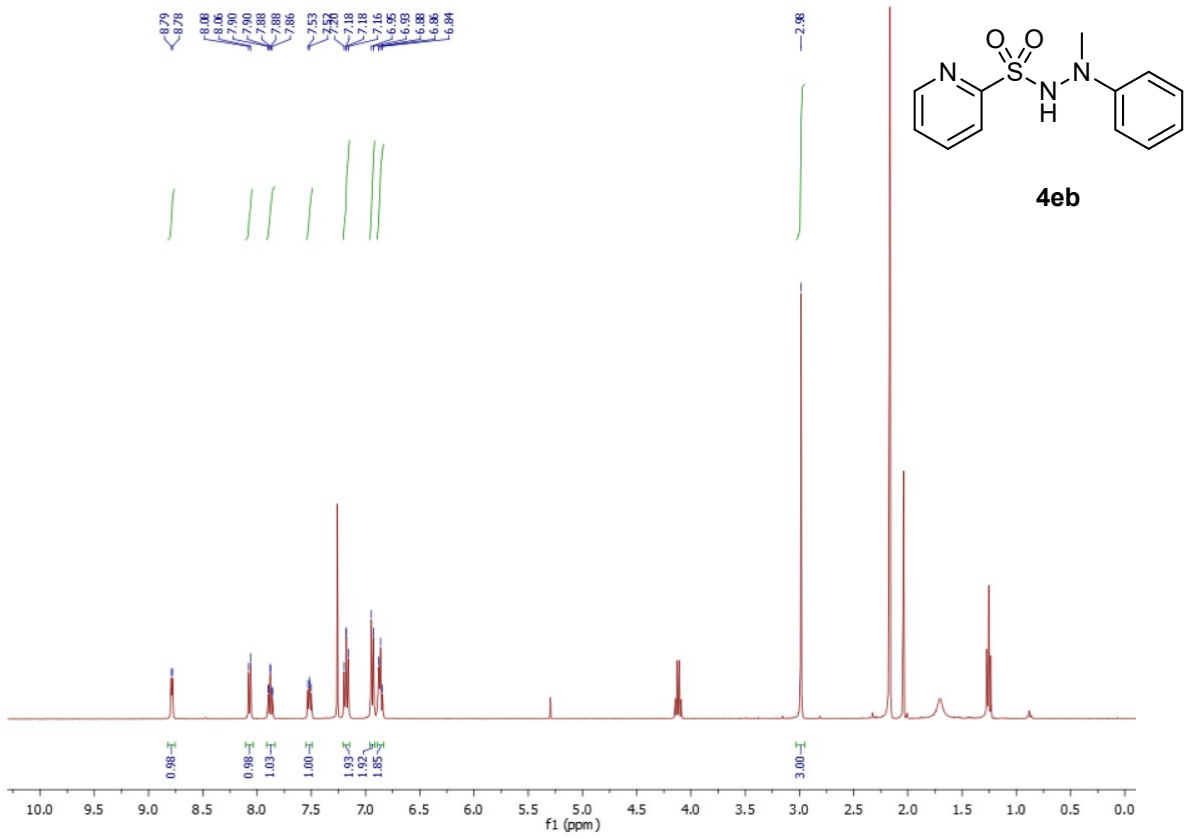
4db

2.16. ^{13}C NMR ($CDCl_3$, 400 MHz) of N'-methyl-N'-phenylnaphthalene-2-sulfonohydrazide (4db)



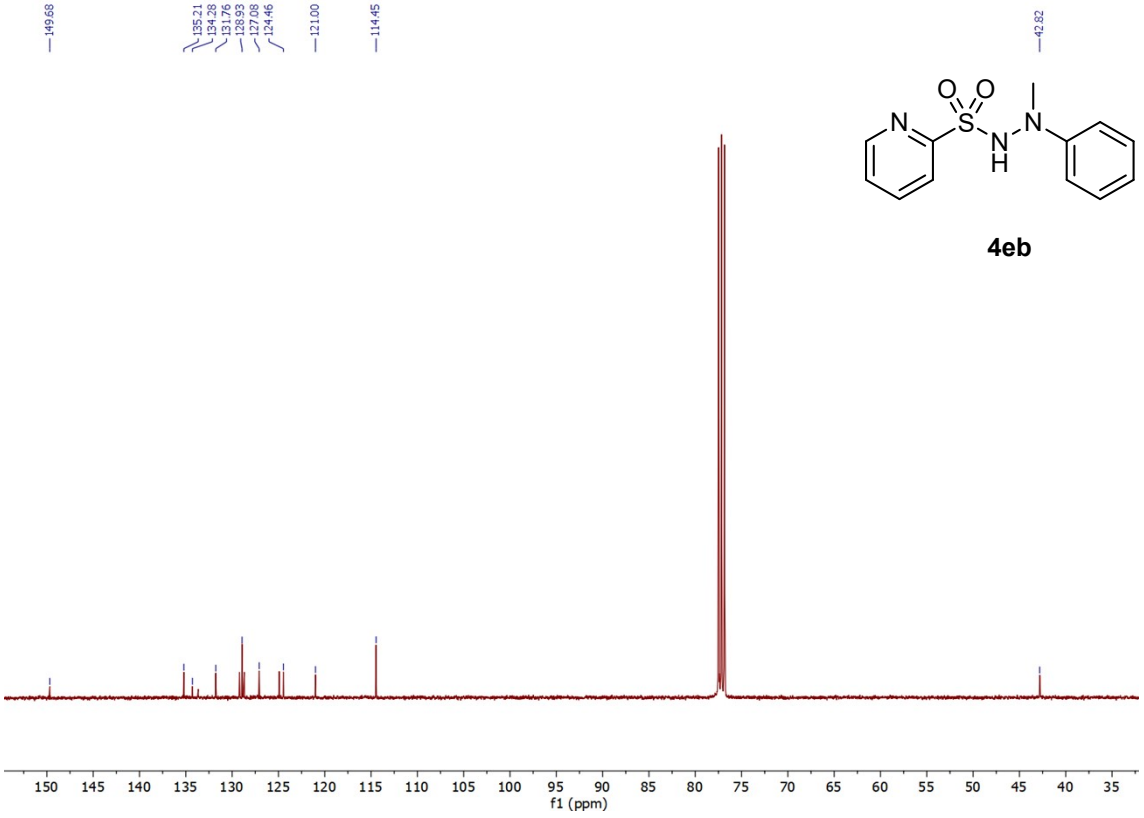
4db

2.17. ^{1}H NMR ($CDCl_3$, 400 MHz) of N'-methyl-N'-phenylpyridin-2-sulfonohydrazide (4eb)



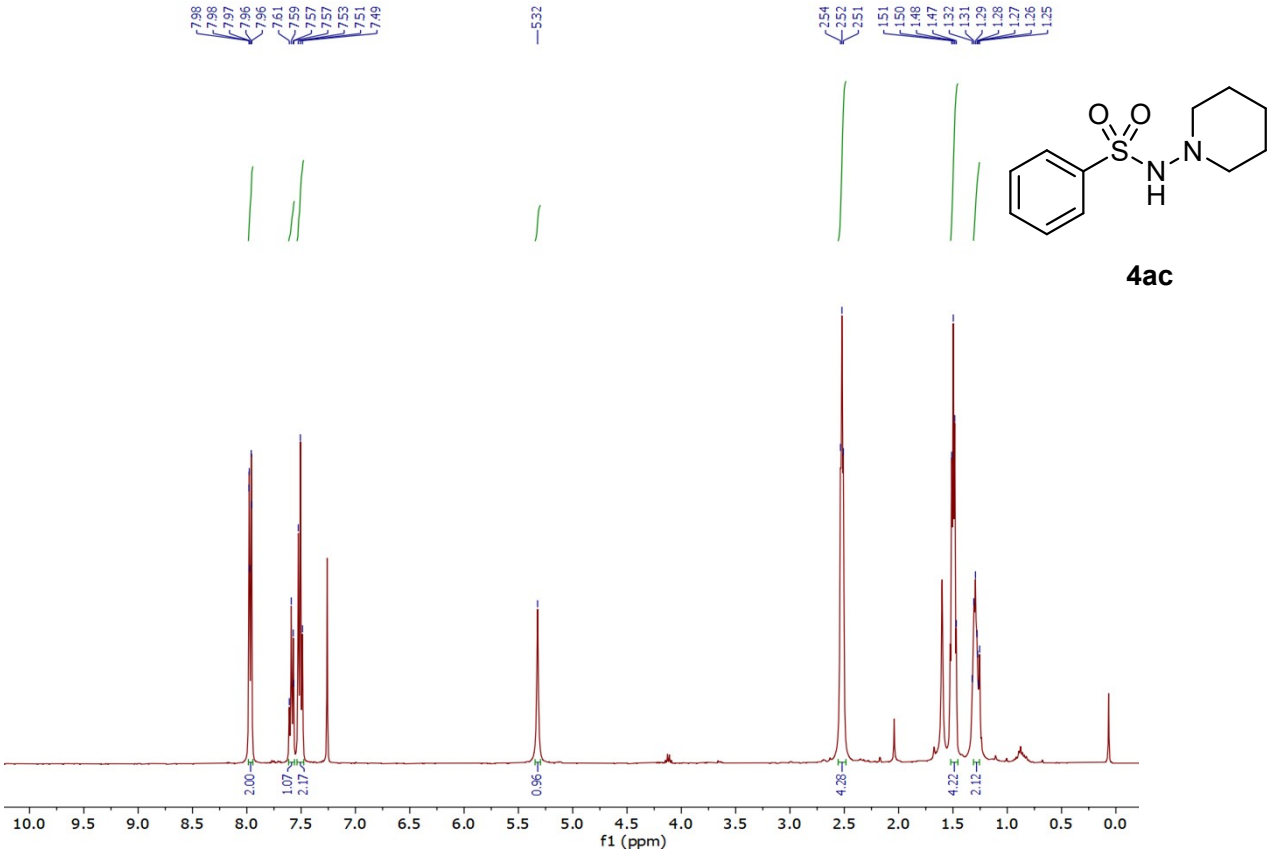
4eb

2.18. ^{13}C NMR ($CDCl_3$, 400 MHz) of N'-methyl-N'-phenylpyridin-2-sulfonohydrazide (4eb)



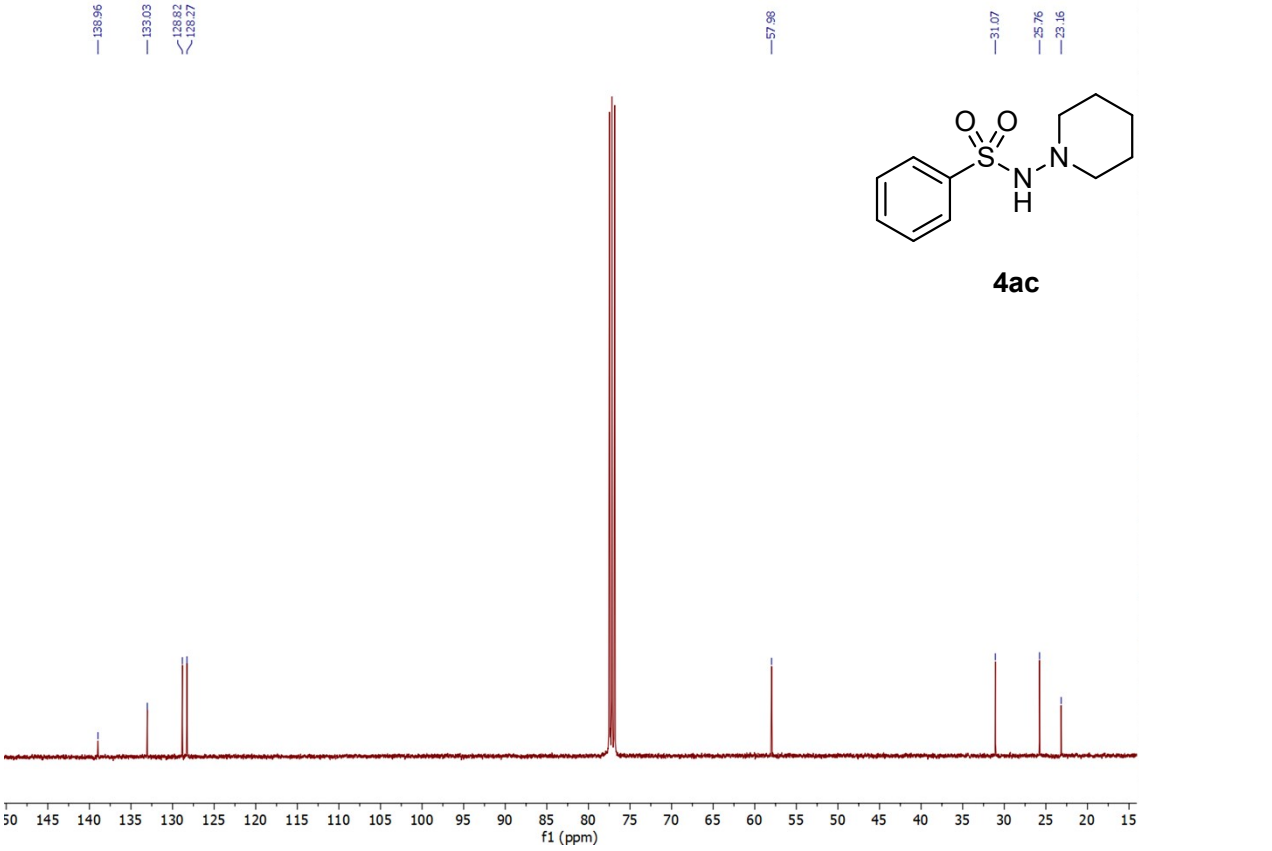
4eb

2.19. ^{1}H NMR ($CDCl_3$, 400 MHz) of N-(piperidin-1-yl)benzenesulfonamide (4ac)



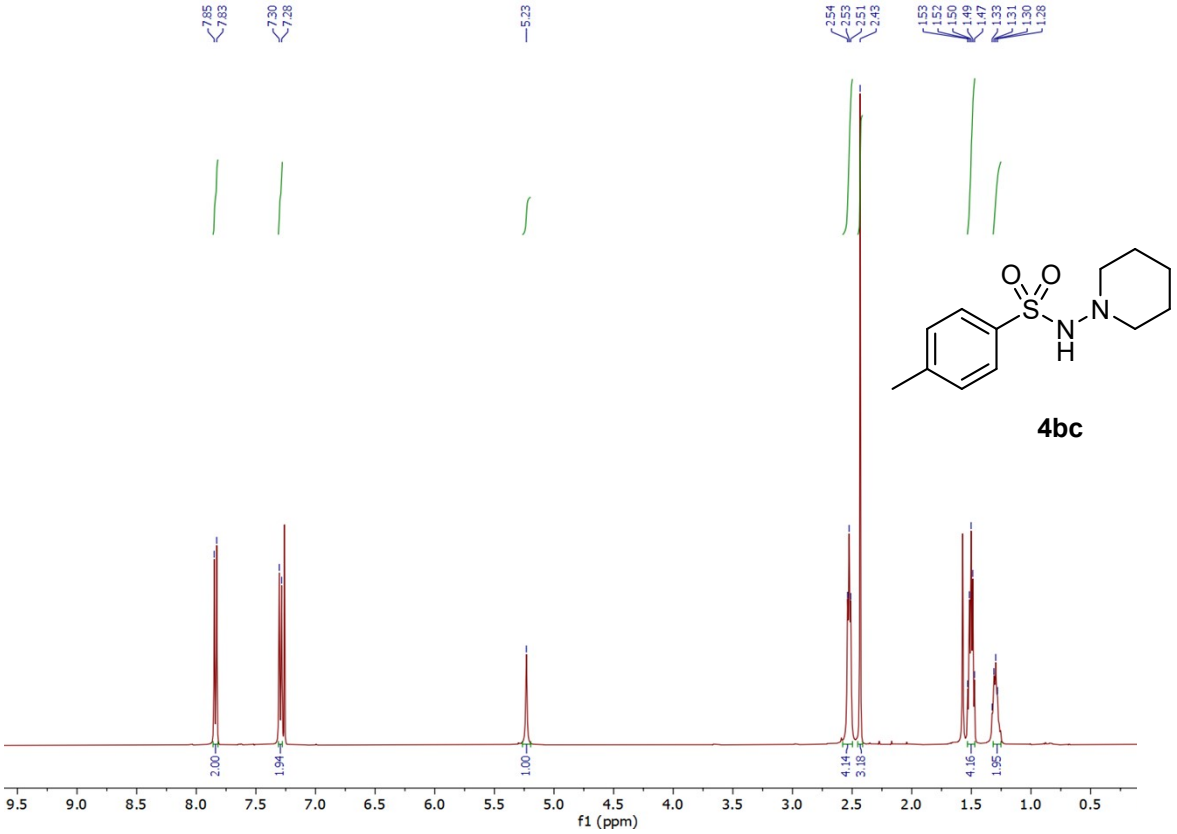
4ac

2.20. ^{13}C NMR ($CDCl_3$, 400 MHz) of N-(piperidin-1-yl)benzenesulfonamide (4ac)

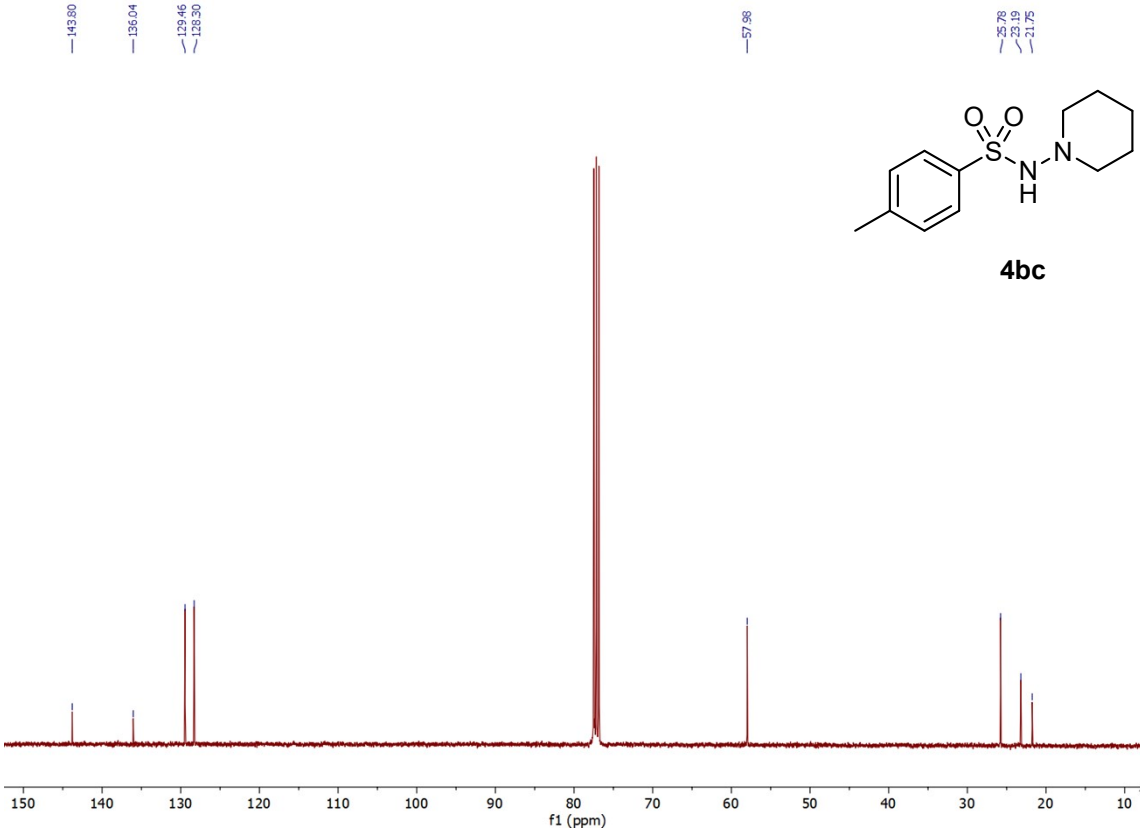


4ac

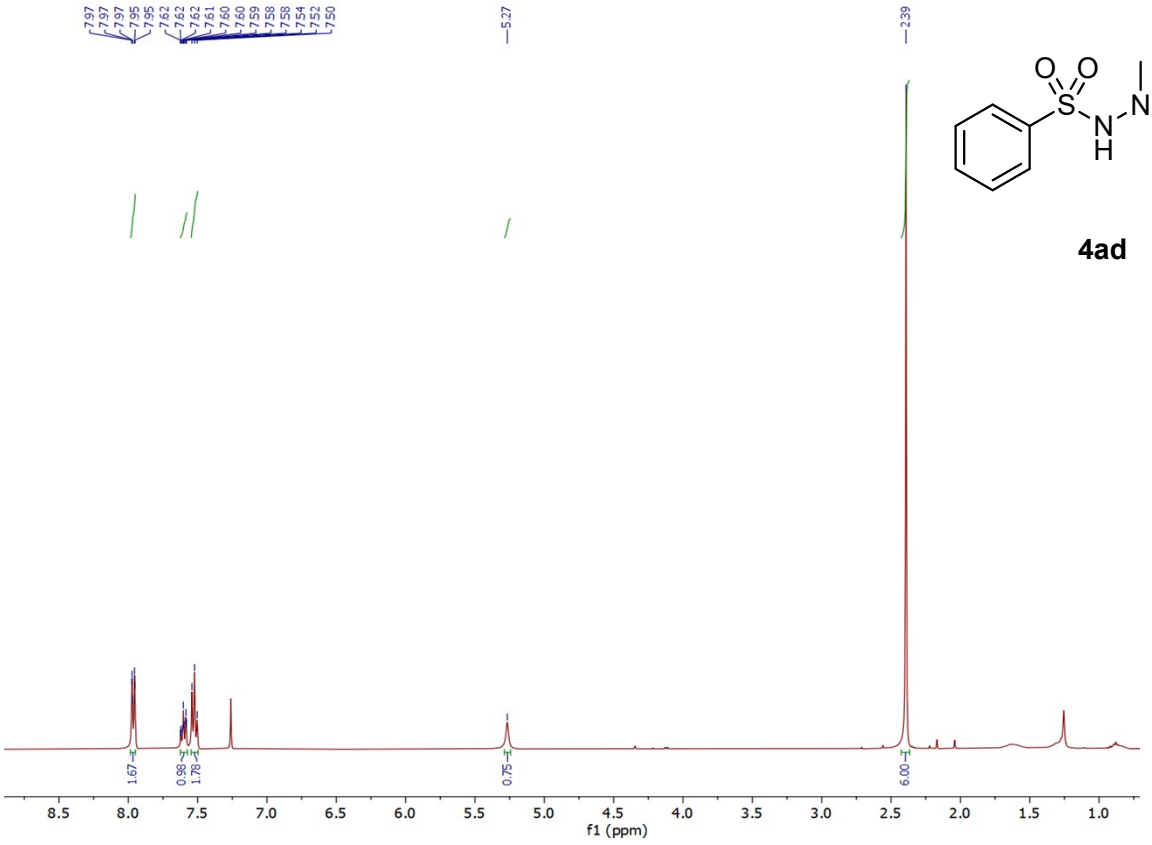
2.21. ^{1}H NMR ($CDCl_3$, 400 MHz) of 4-methyl-N-(piperidin-1 yl) benzenesulfonamide (4bc)



2.22. ^{13}C NMR ($CDCl_3$, 400 MHz) of 4-methyl-N-(piperidin-1 yl) benzenesulfonamide (4bc)

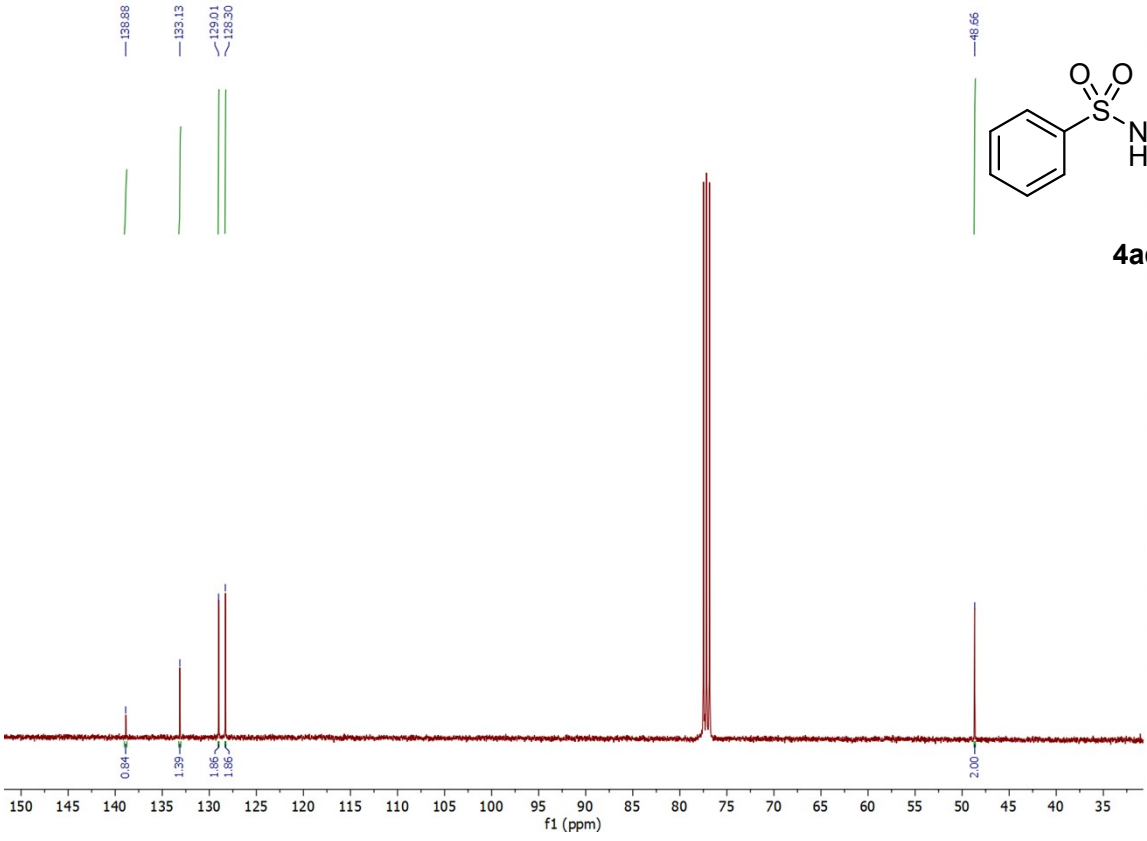


2.23. ^{1}H NMR ($CDCl_3$, 400 MHz) of 2-Benzolsulfonyl-1,1-dimethyl-hydrazin (4ad)



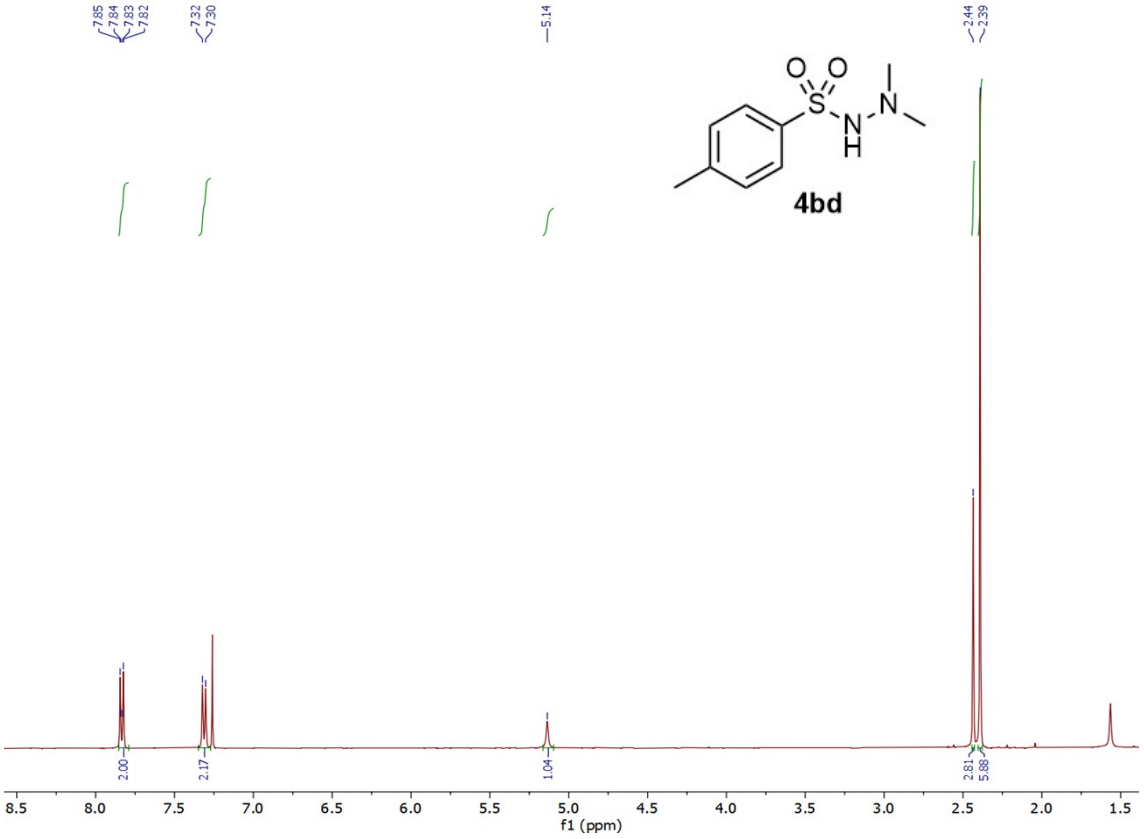
4ad

2.24. ^{13}C NMR ($CDCl_3$, 400 MHz) of 2-Benzolsulfonyl-1,1-dimethyl-hydrazin (4ad)

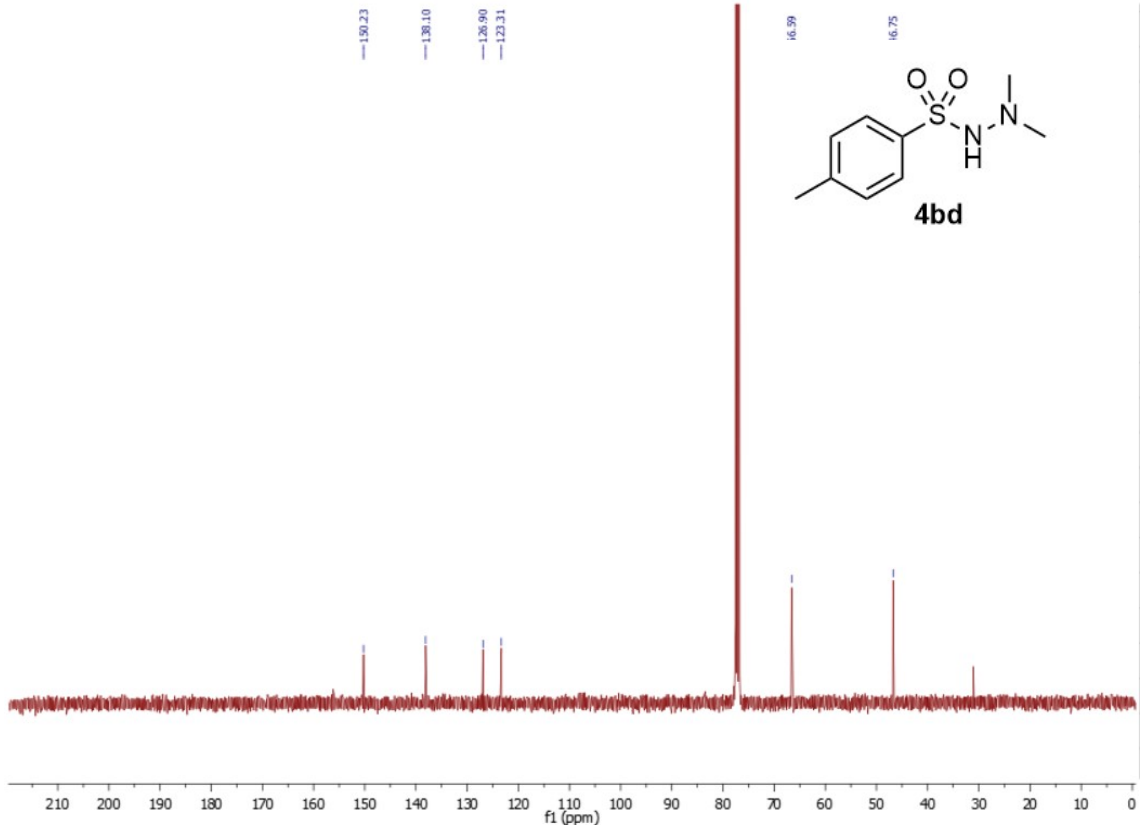


4ad

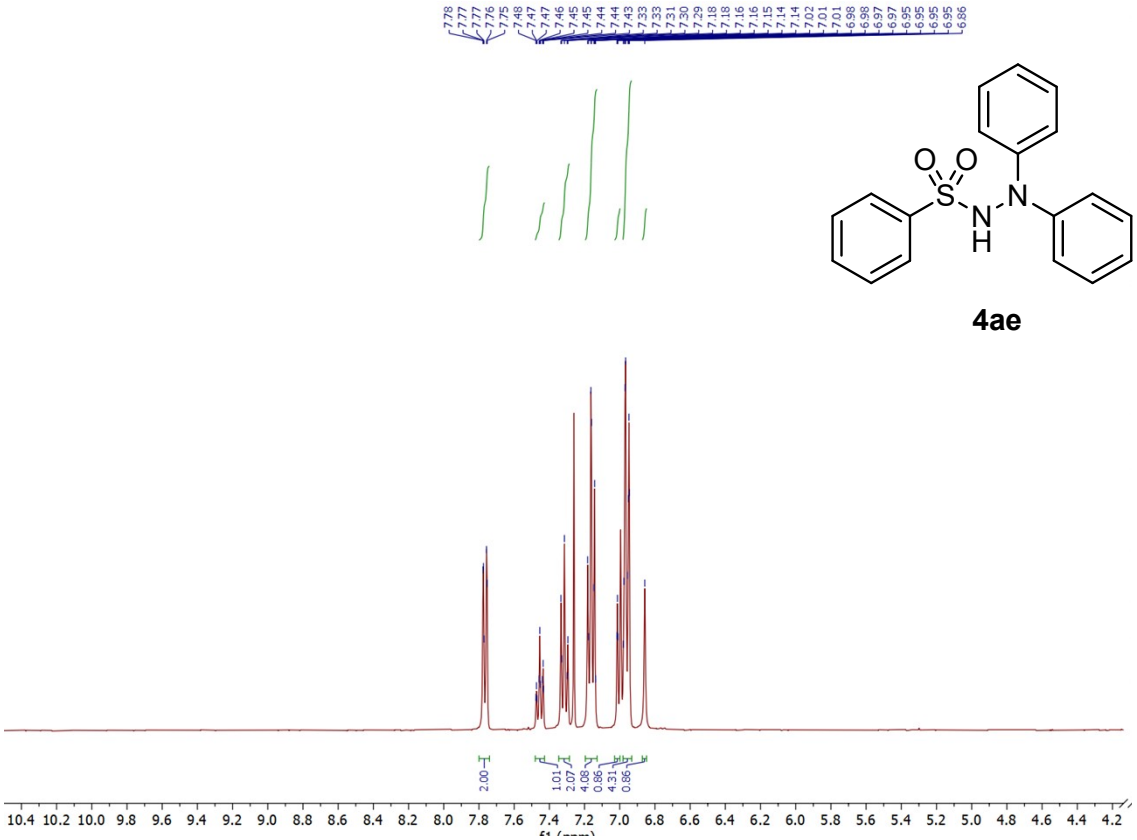
2.25. ^{1}H NMR ($CDCl_3$, 400 MHz) of N,N-Dimethyl-4-toluenesulfonylhydrazide (4bd)



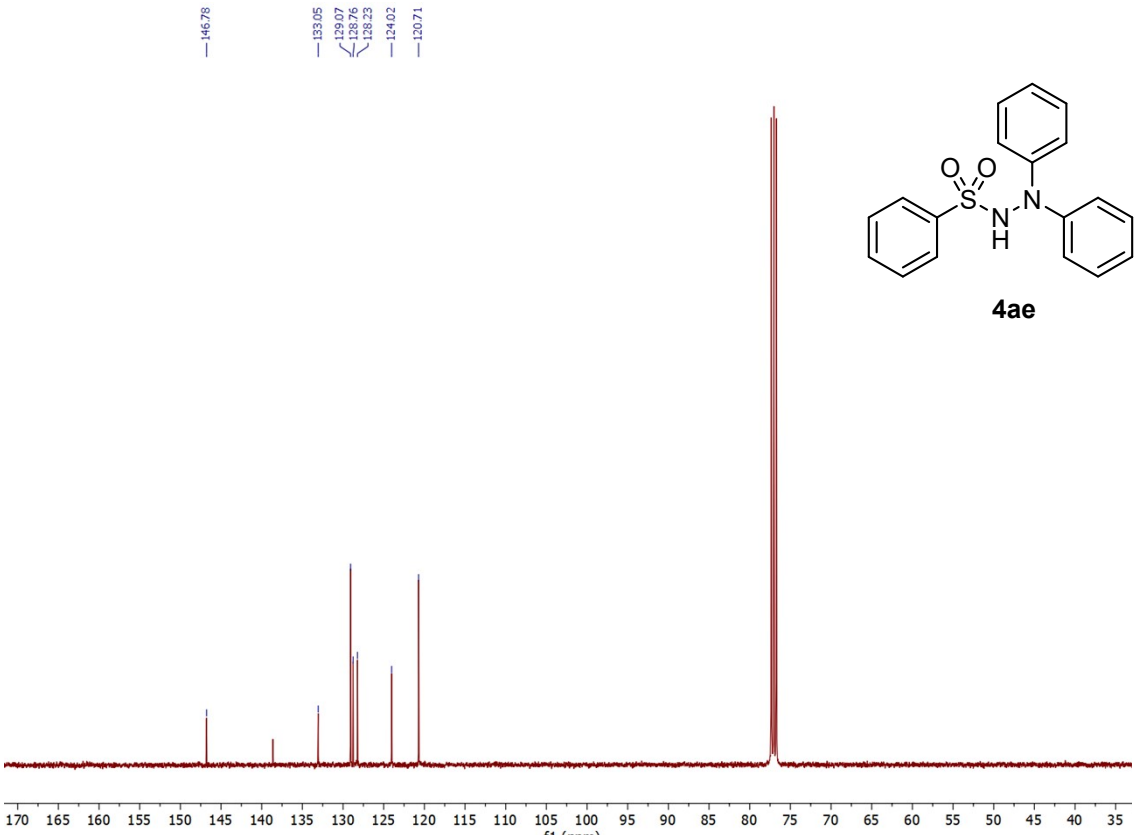
2.26. ^{13}C NMR ($CDCl_3$, 400 MHz) of N,N-Dimethyl-4-toluenesulfonylhydrazide (4bd)



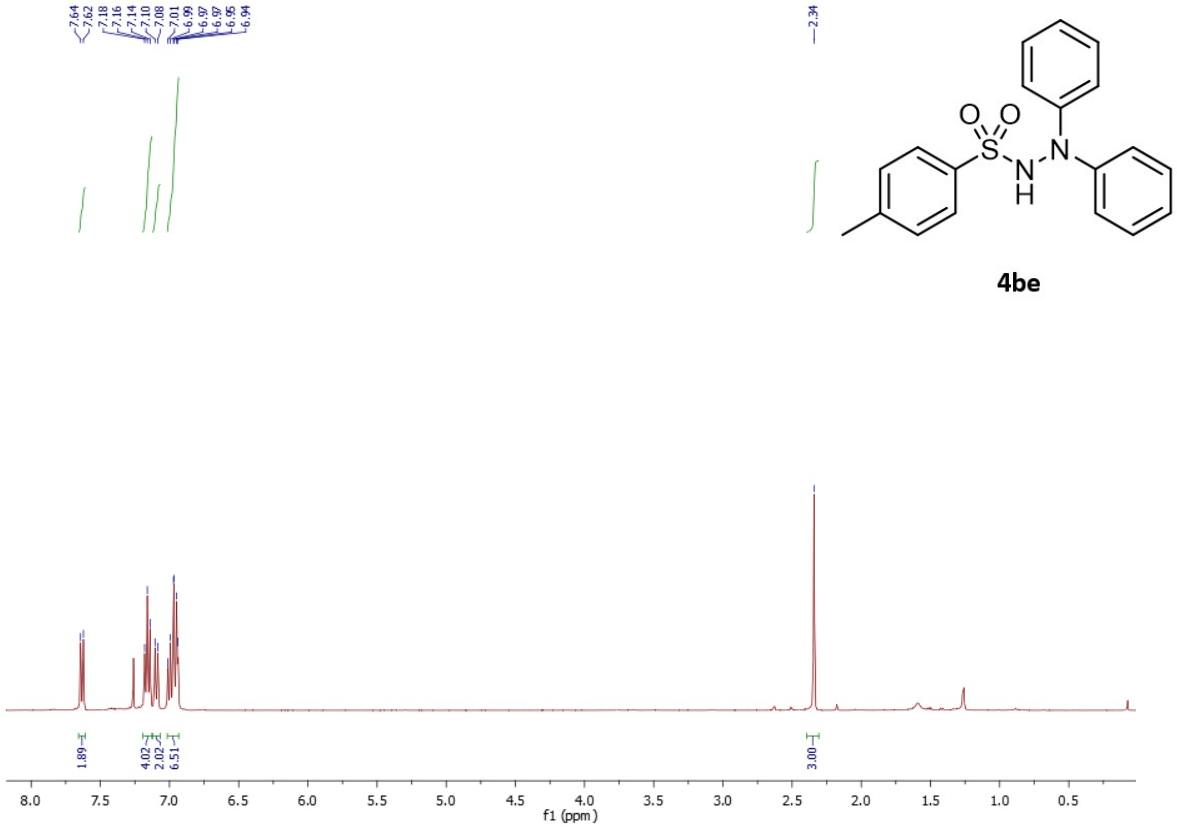
2.27. ^{1}H NMR ($CDCl_3$, 400 MHz) of N',N'-diphenylbenzenesulfonohydrazide (4ae)



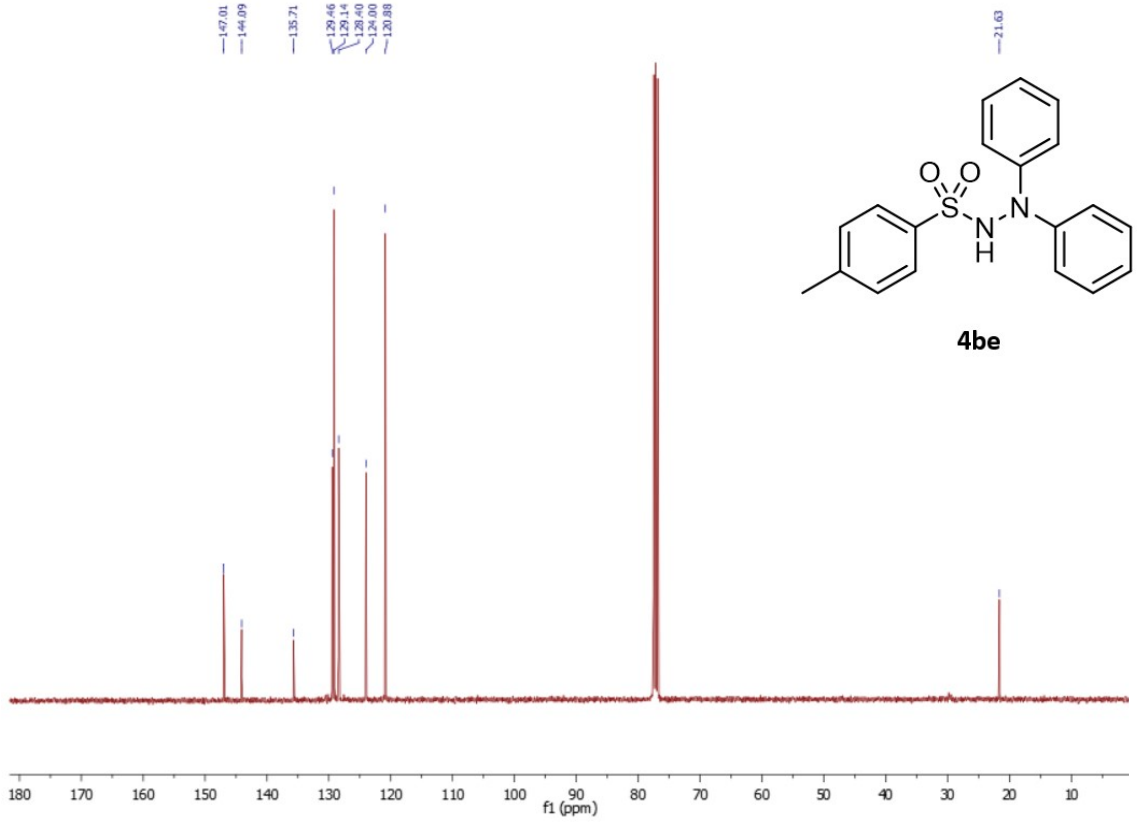
2.28. ^{13}C NMR ($CDCl_3$, 400 MHz) of N',N'-diphenylbenzenesulfonohydrazide (4ae)



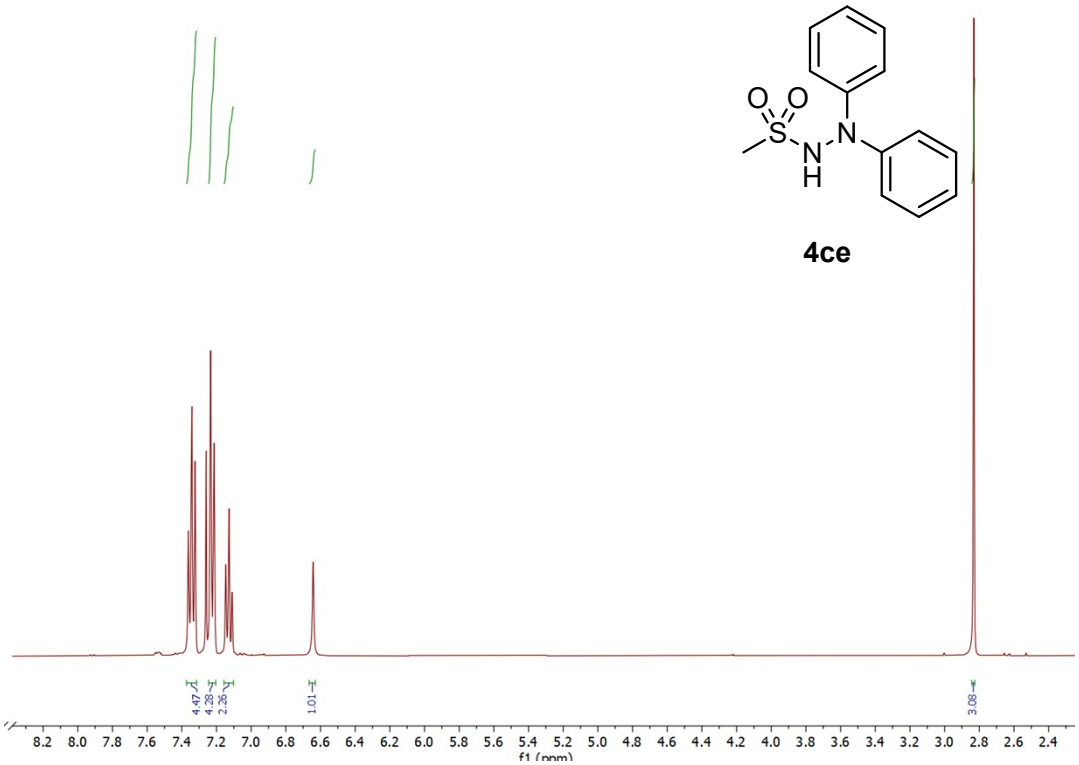
2.29. ^{1}H NMR ($CDCl_3$, 400 MHz) of 4-methyl-N',N'diphenylbenzene sulfonylhydrazide (4be)



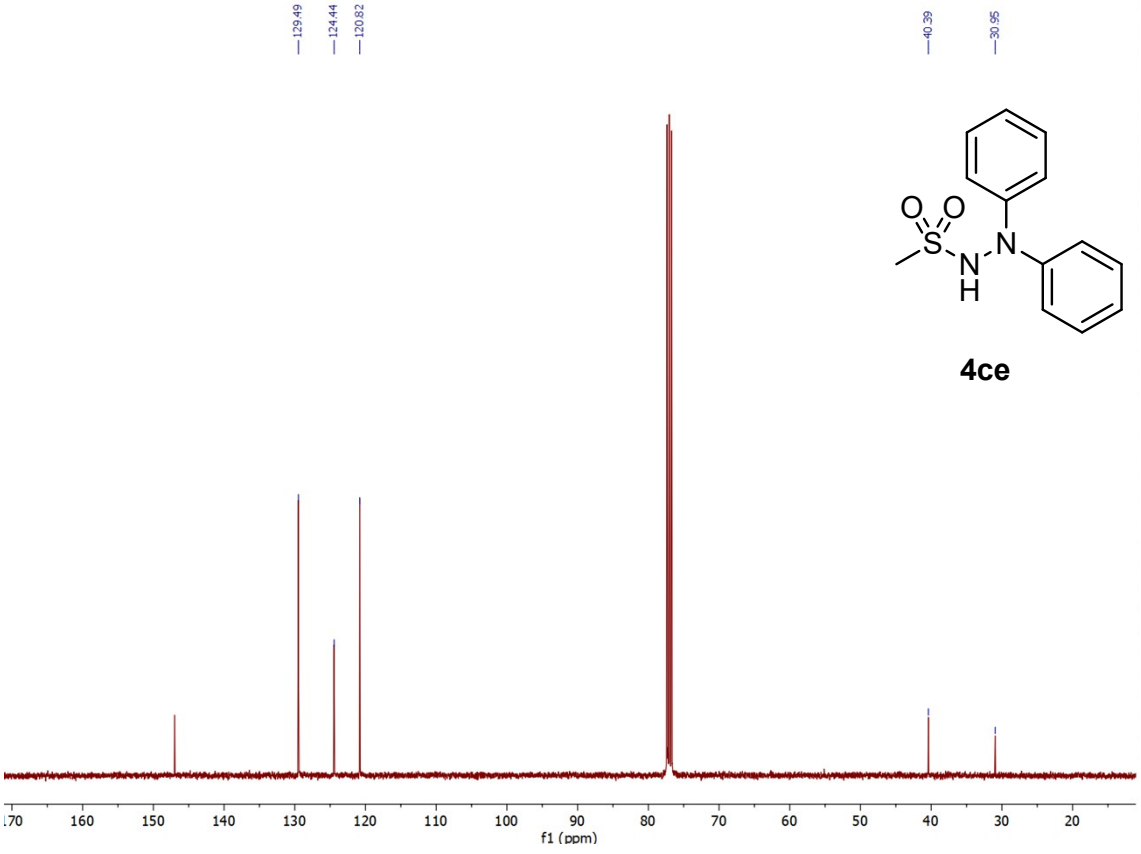
2.30. ^{13}C NMR ($CDCl_3$, 400 MHz) of 4-methyl-N',N'diphenylbenzene sulfonylhydrazide (4be)



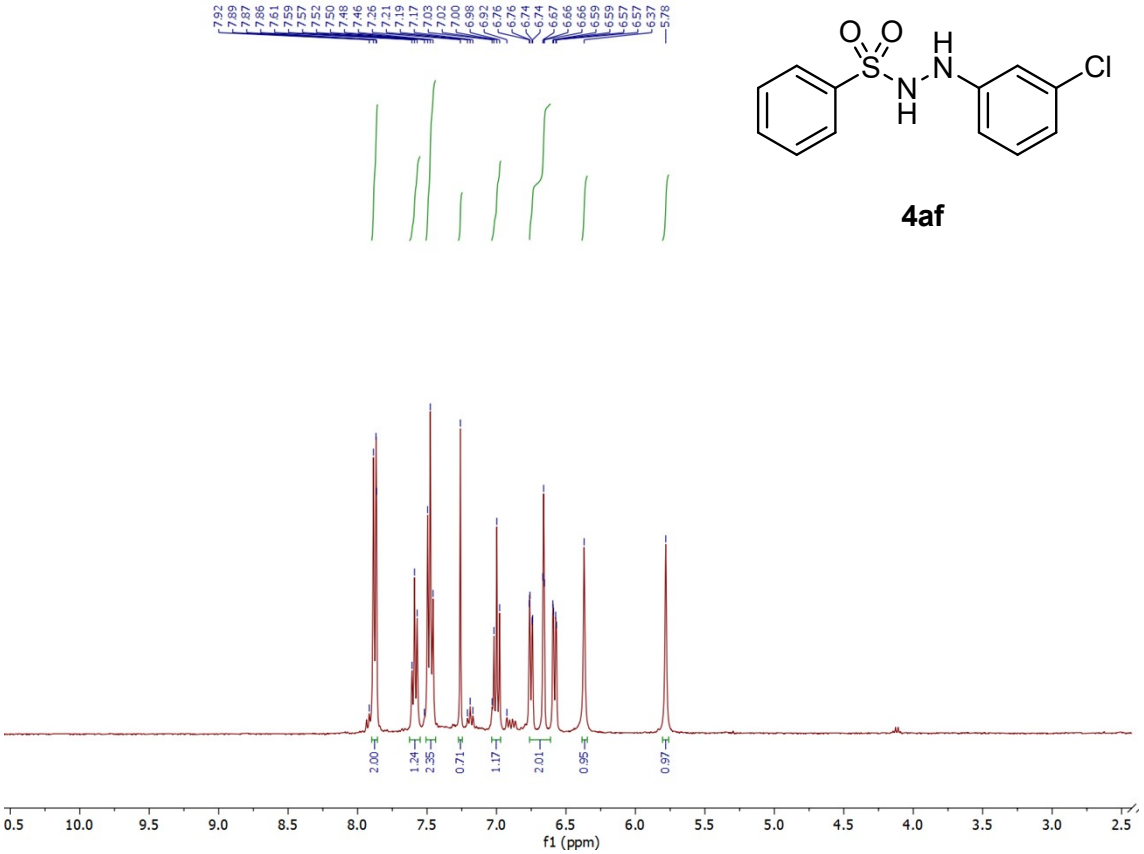
2.31. ^{1}H NMR ($CDCl_3$, 400 MHz) of 1-methanesulfonyl-2-phenylhydrazine (4ce)



2.32. ^{13}C NMR ($CDCl_3$, 400 MHz) of 1-methanesulfonyl-2-phenylhydrazine (4ce)

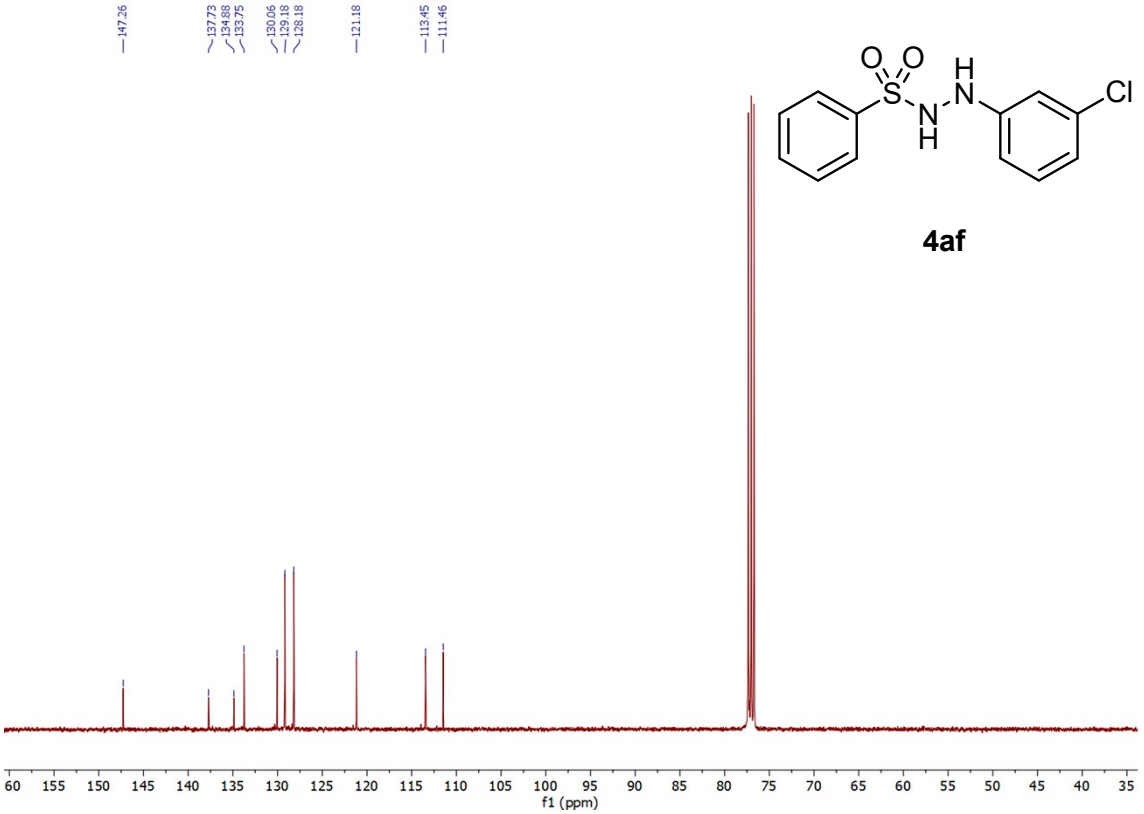


2.33. ^{1}H NMR ($CDCl_3$, 400 MHz) of N'-(3-chlorophenyl)benzenesulfonohydrazide (4af)



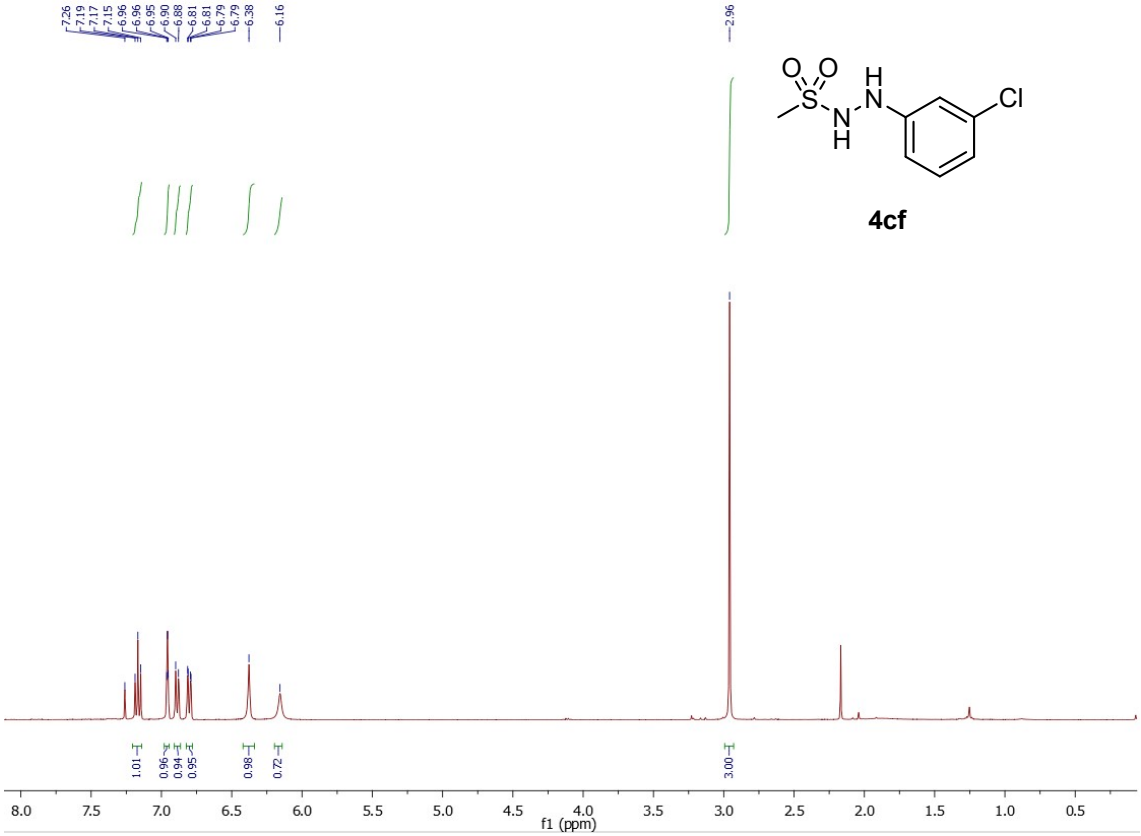
4af

2.34. ^{13}C NMR ($CDCl_3$, 400 MHz) of N'-(3-chlorophenyl)benzenesulfonohydrazide (4af)

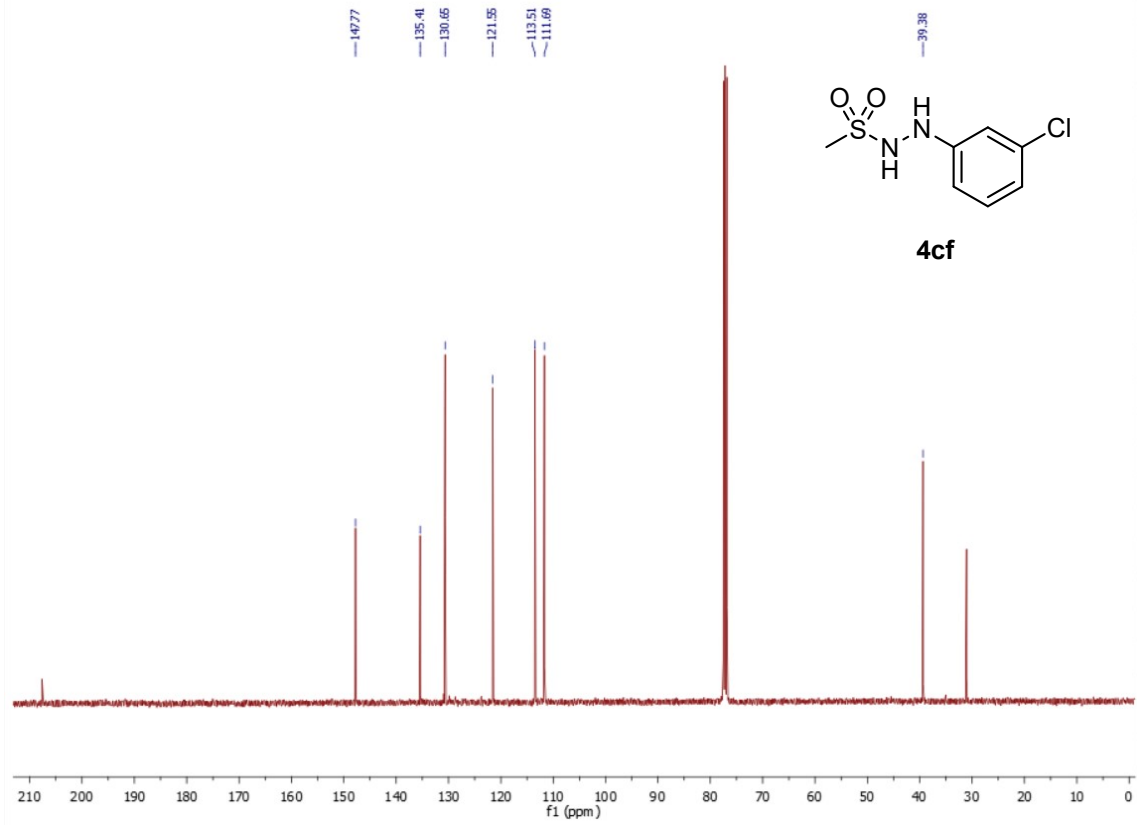


4af

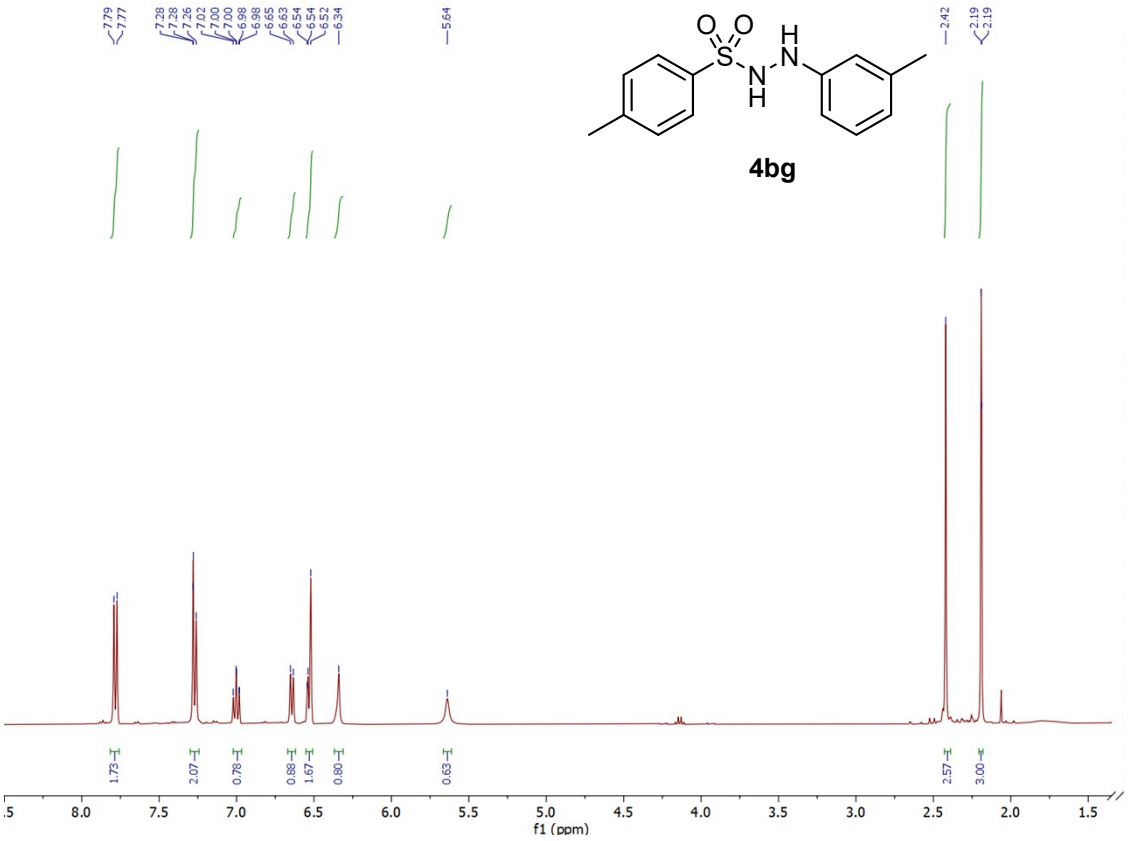
2.35. ^{1}H NMR ($CDCl_3$, 400 MHz) of N'-(3-chlorophenyl)methanesulfonohydrazide (4cf)



2.36. ^{13}C NMR ($CDCl_3$, 400 MHz) of N'-(3-chlorophenyl)methanesulfonohydrazide (4cf)

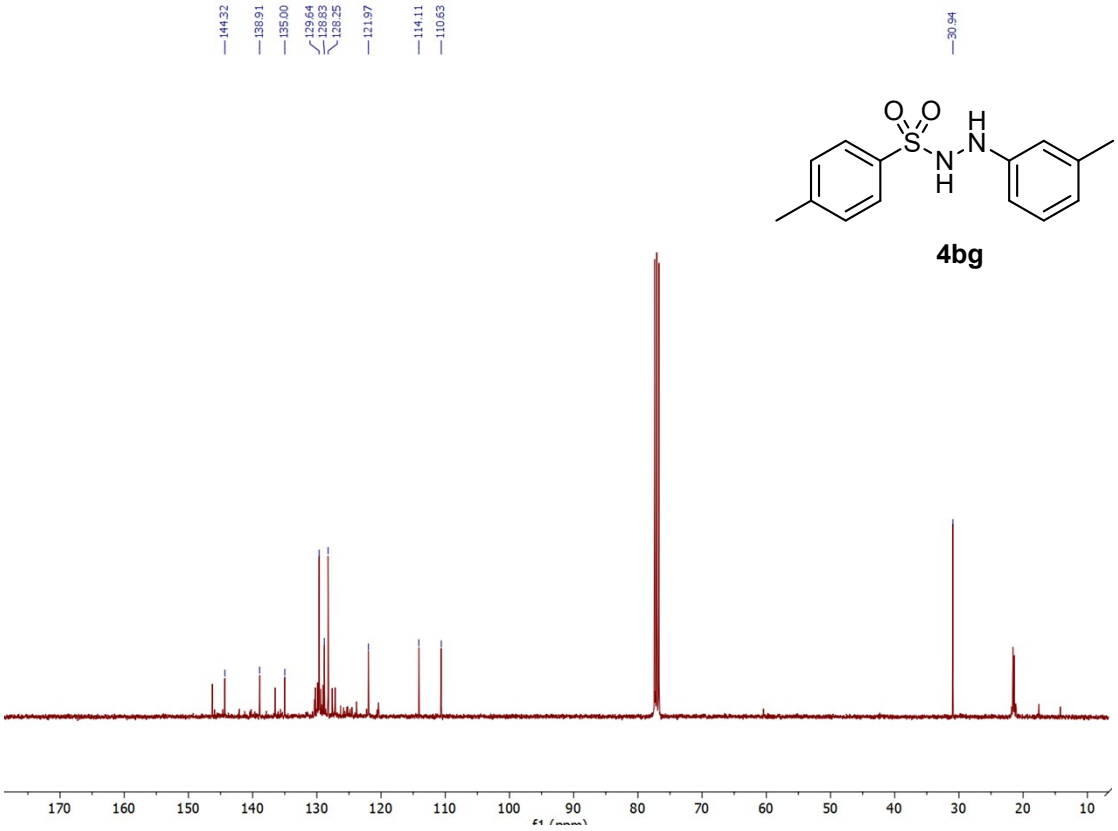


2.37. ^{1}H NMR ($CDCl_3$, 400 MHz) of 4-methyl-N'-(m-tolyl)benzene sulfonohydrazide (4bg)



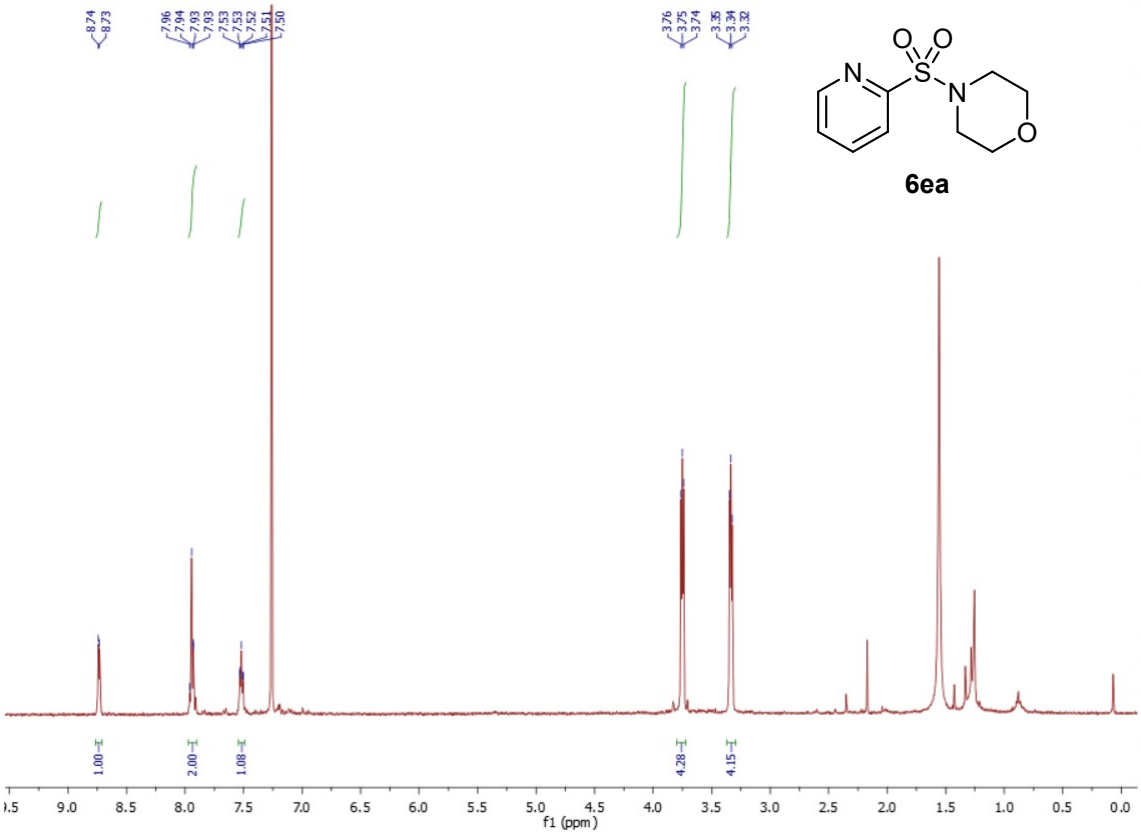
4bg

2.38. ^{13}C NMR ($CDCl_3$, 400 MHz) of 4-methyl-N'-(m-tolyl)benzene sulfonohydrazide (4bg)



4bg

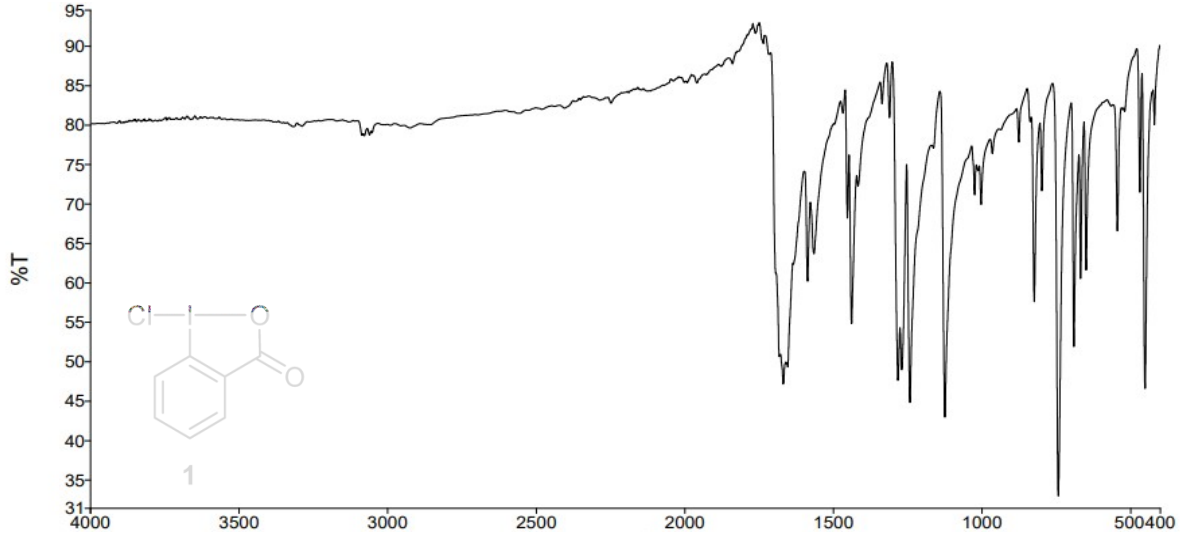
2.39. ^{1}H NMR ($CDCl_3$, 400 MHz) of 4-(pyridin-2-ylsulfonyl)morpholine (6ea)



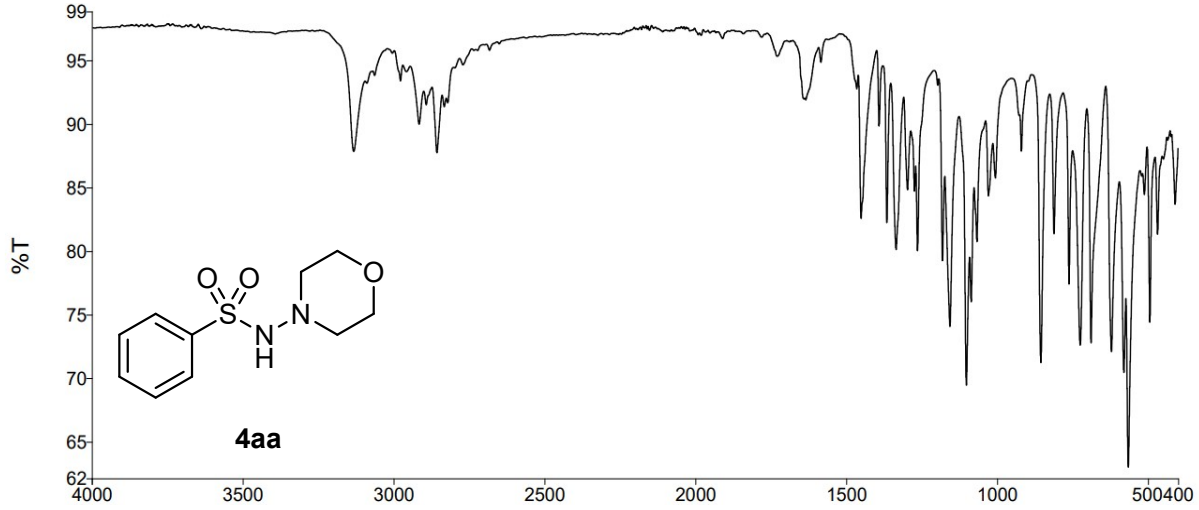
6ea

3. IR Spectra

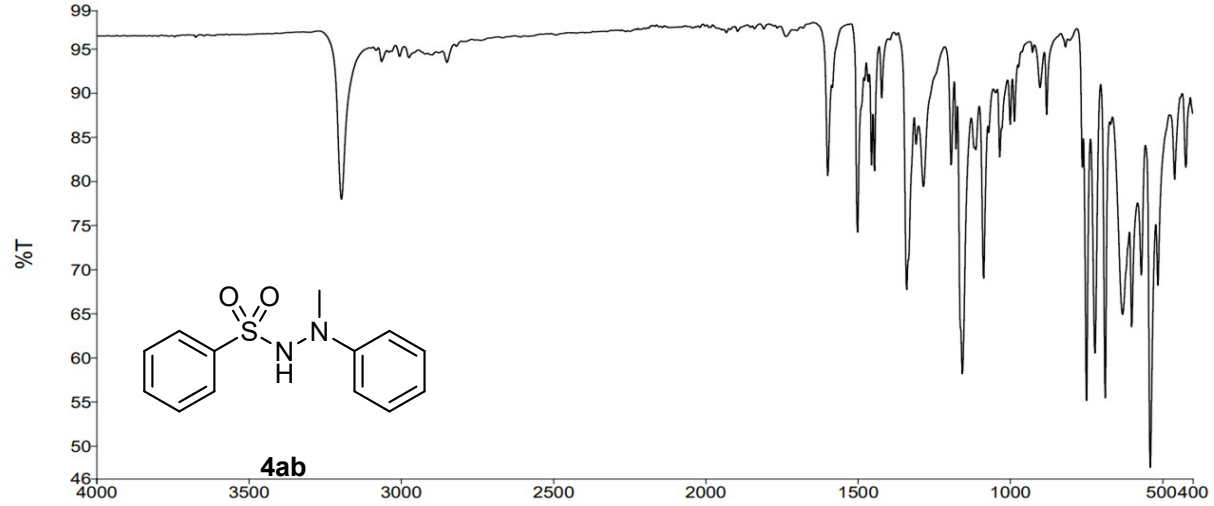
3.1. IR spectra of 1-chloro-1,2-benziodoxol-3-(1H)-one (1)



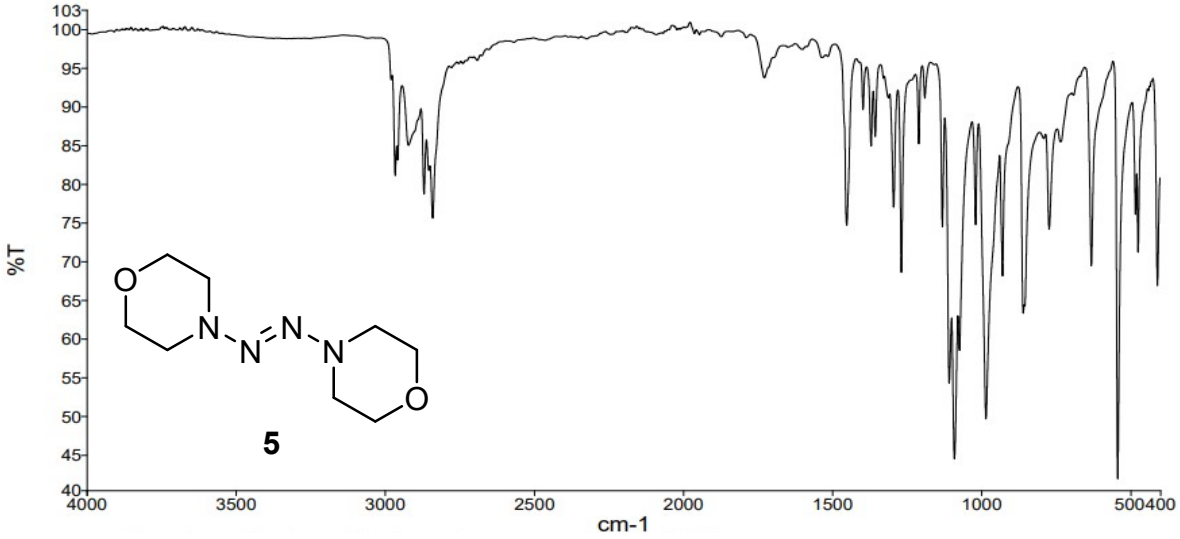
3.2. IR spectra of N-morpholinobenzenesulfonamide (4aa)



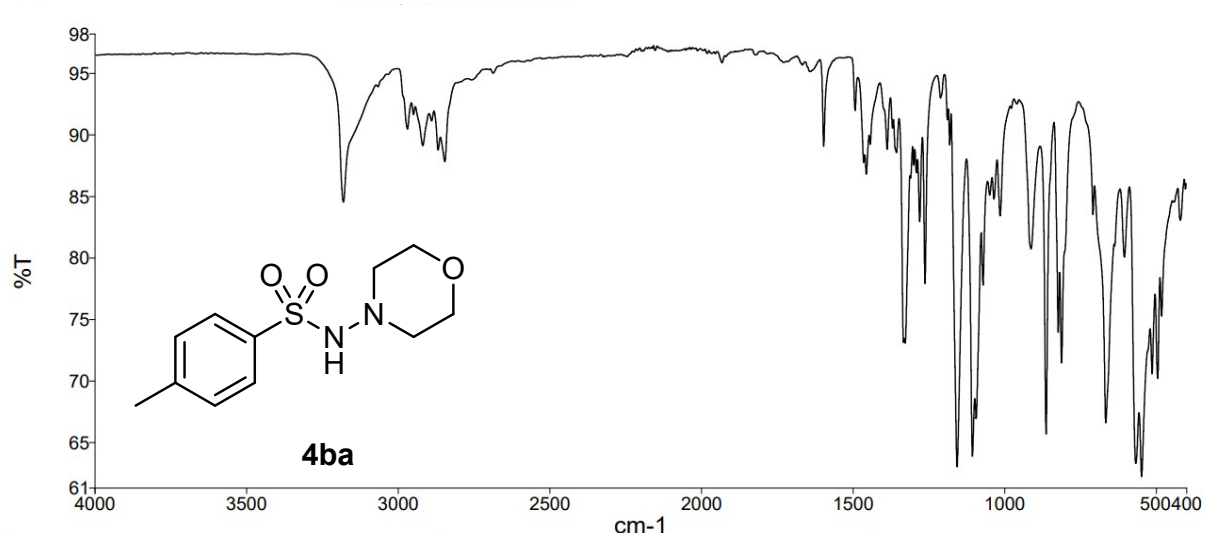
3.3. IR spectra of N'-methyl-N'-phenylbenzenesulfonohydrazide (4ab)



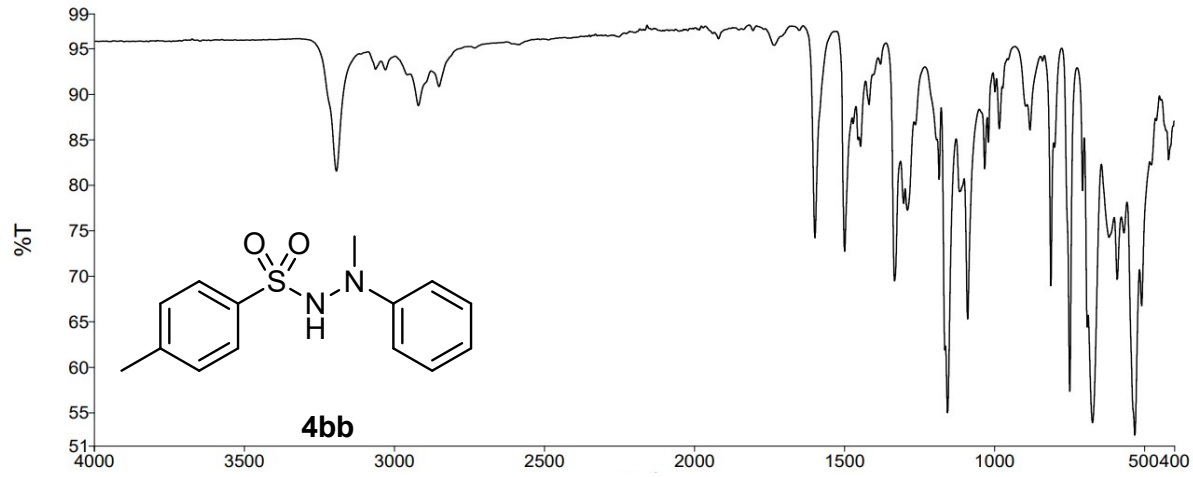
3.4. IR spectra of 1,2-dimorpholinodiazene (5)



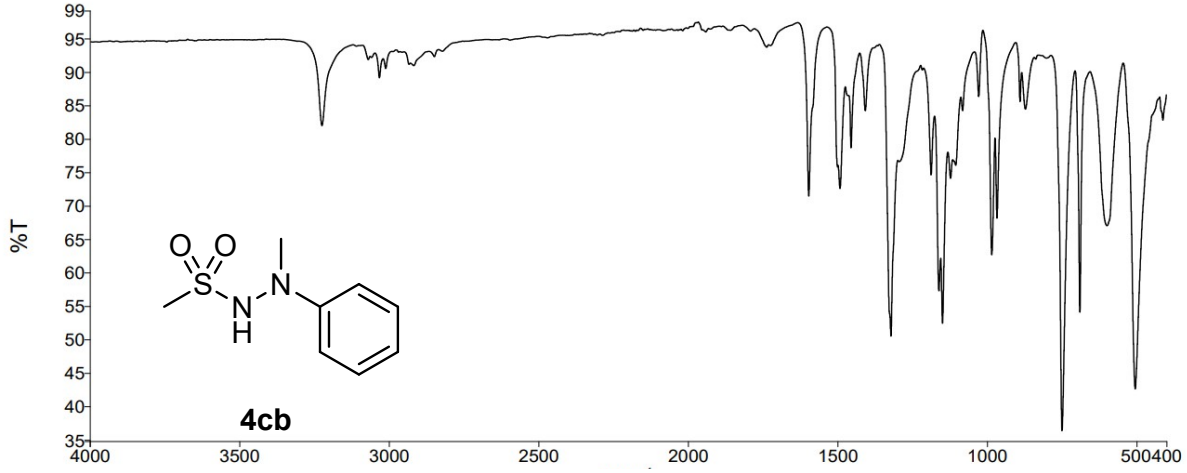
3.5. IR spectra of N-morpholinobenzenesulfonamide (4ba)



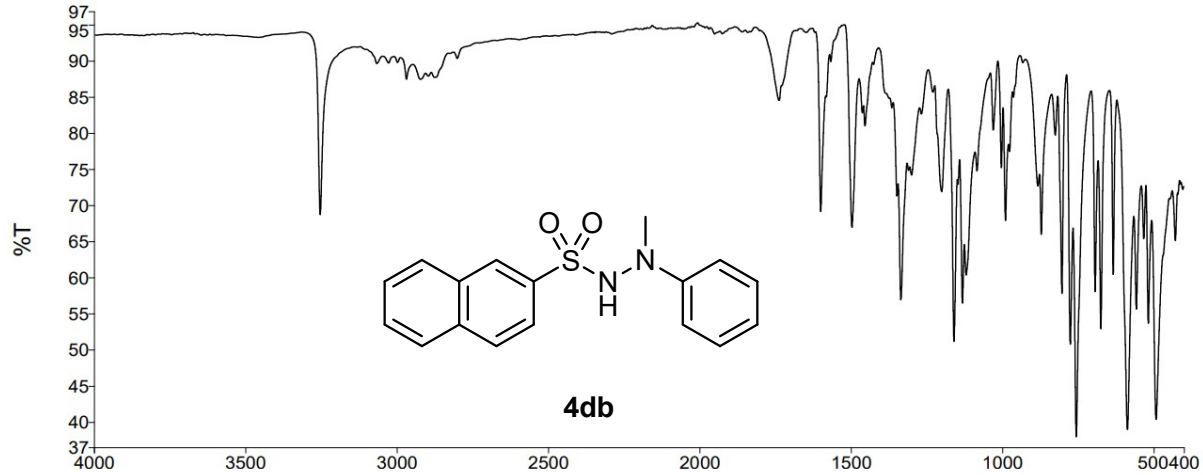
3.6. IR spectra of N’-Methyl-N-phenyl-N'-p-toluolsulfonyl-hydrazin (4bb)



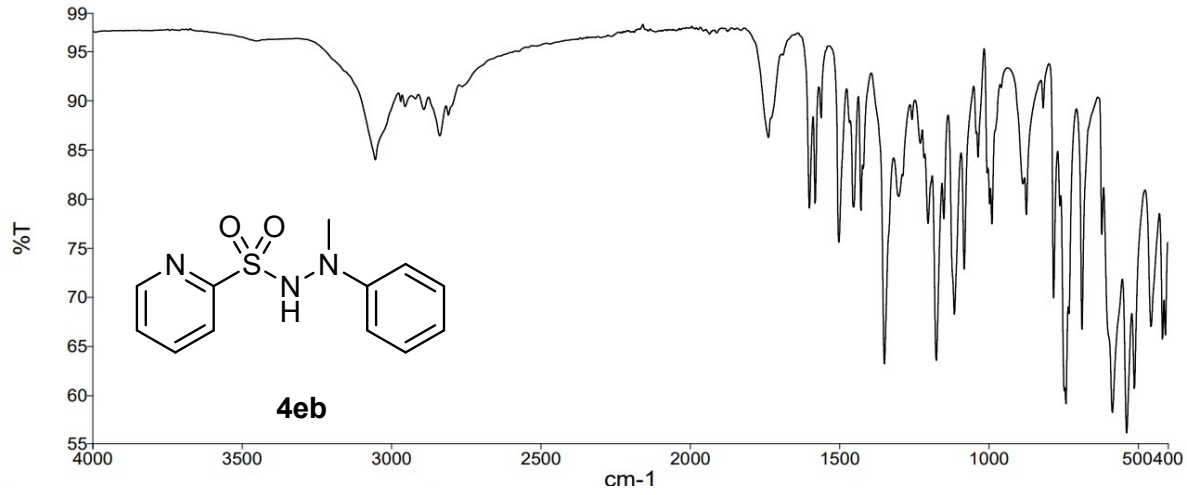
3.7. IR spectra of N'-methyl-N'-phenylmethanesulfonohydrazide (4cb)



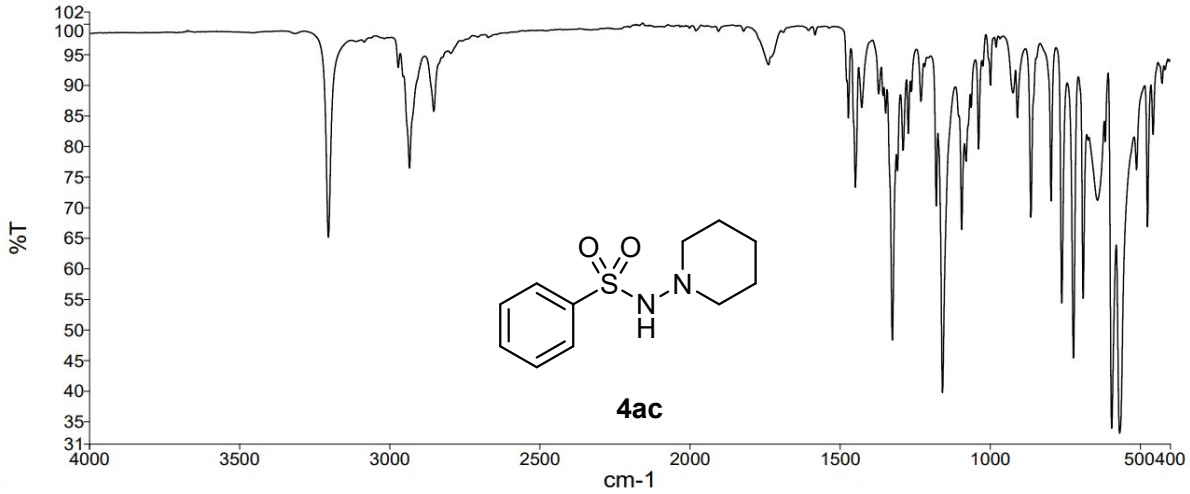
3.8. IR spectra of N'-methyl-N'-phenylnaphthalene-2-sulfonohydrazide (4db)



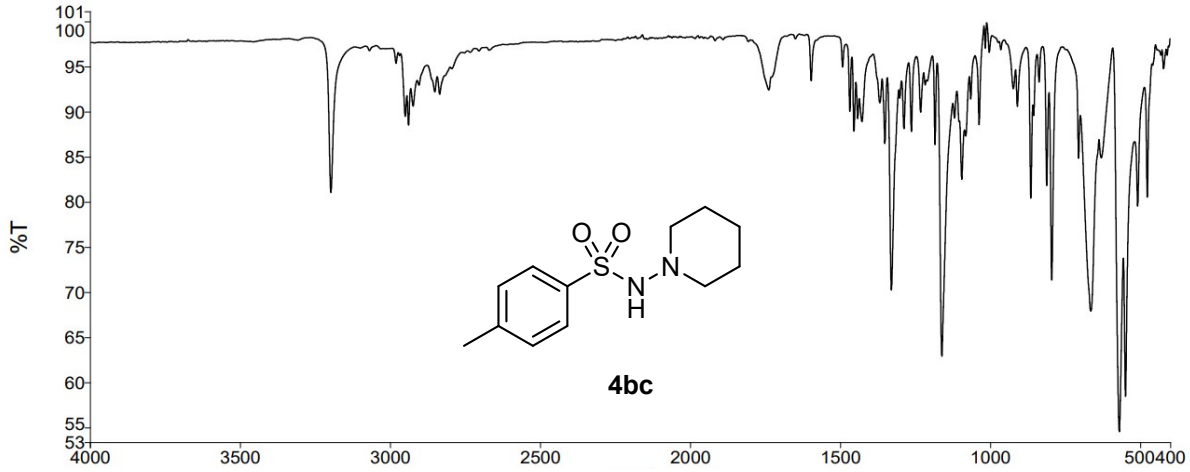
3.9. IR spectra of N'-methyl-N'-phenylpyridin-2-sulfonohydrazide (4eb)



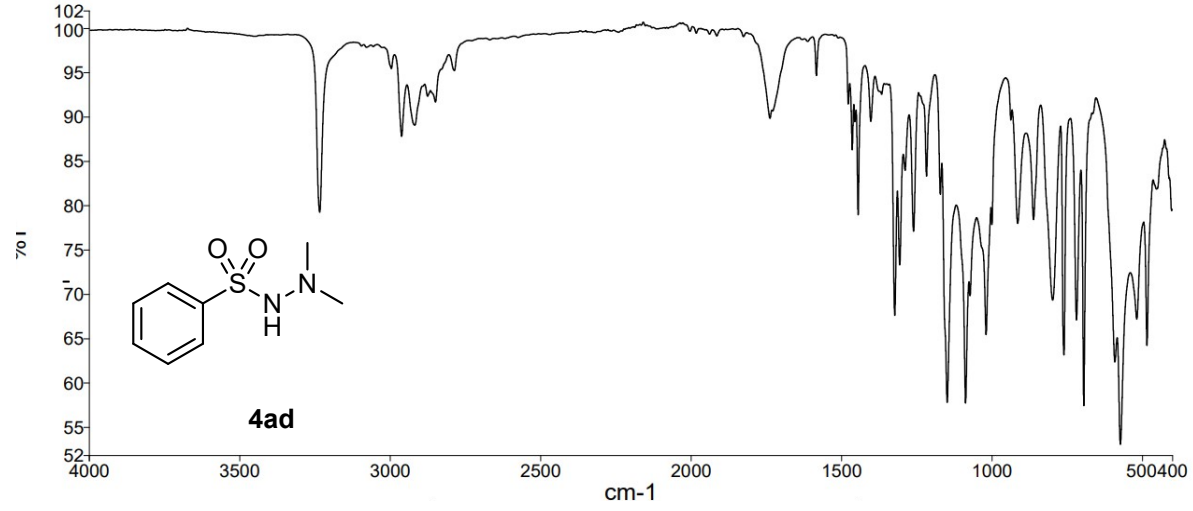
3.10. IR spectra of N-(piperidin-1-yl)benzenesulfonamide (4ac)



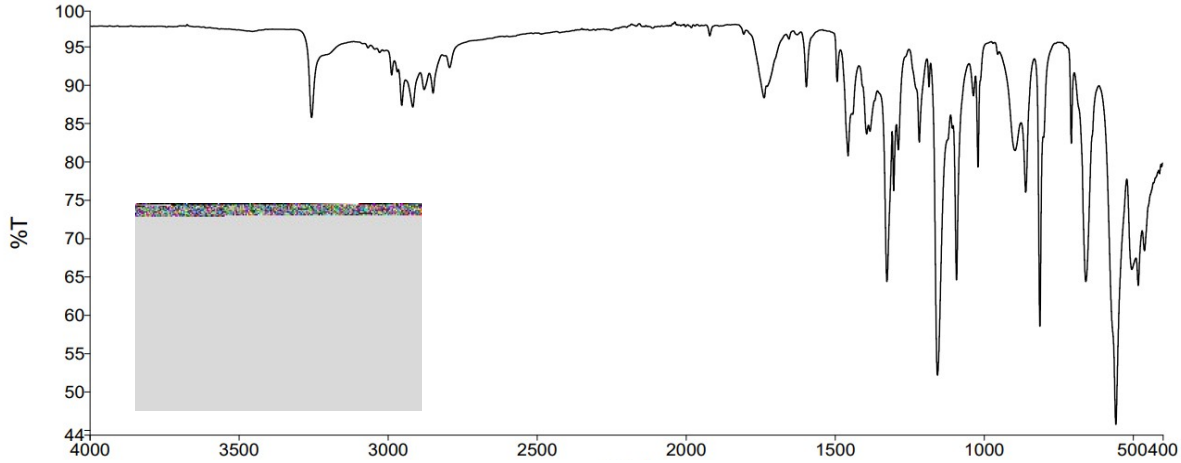
3.11. IR spectra of 4-methyl-N-(piperidin-1 yl) benzenesulfonamide (4bc)



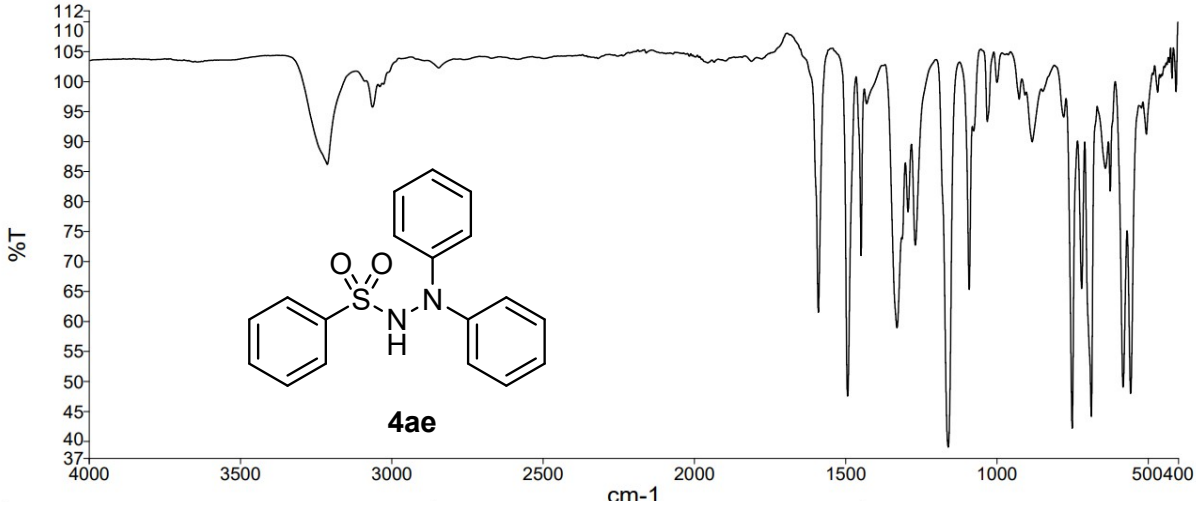
3.12. IR spectra of 2-Benzolsulfonyl-1,1-dimethyl-hydrazin (4ad)



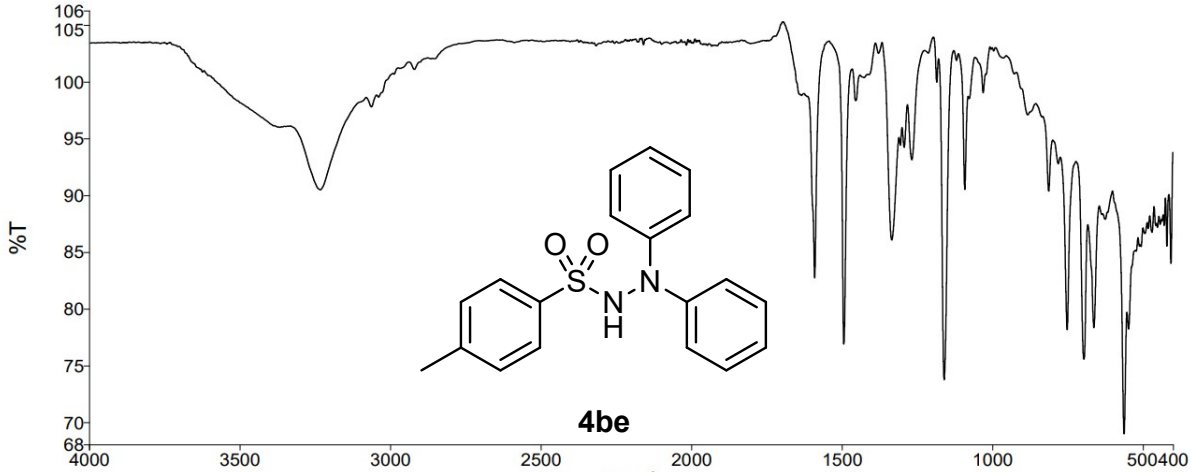
3.13. IR spectra of N,N-Dimethyl-4-toluenesulfonylhydrazide (4bd)



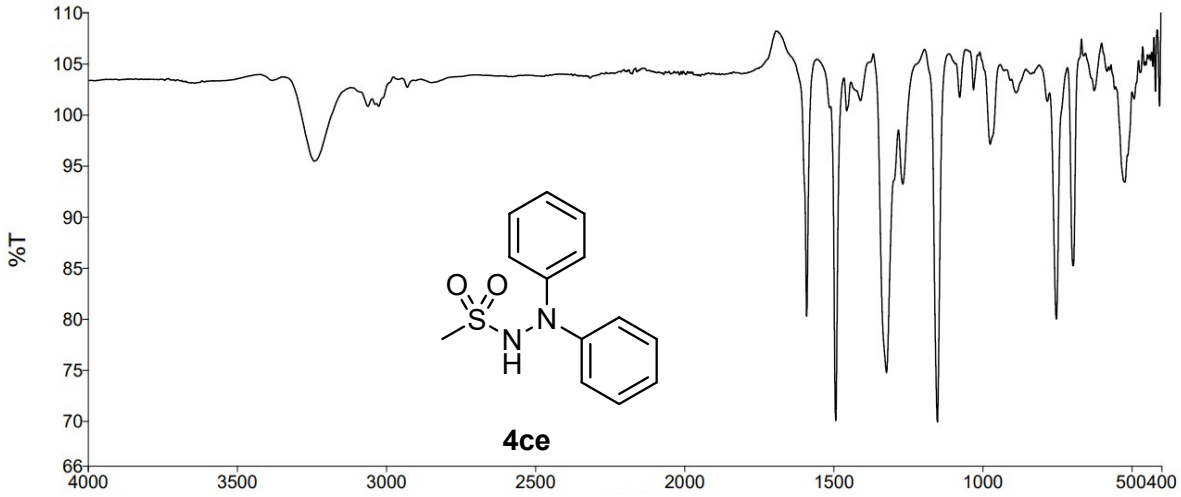
3.14. IR spectra of N',N'-diphenylbenzenesulfonohydrazide (4ae)



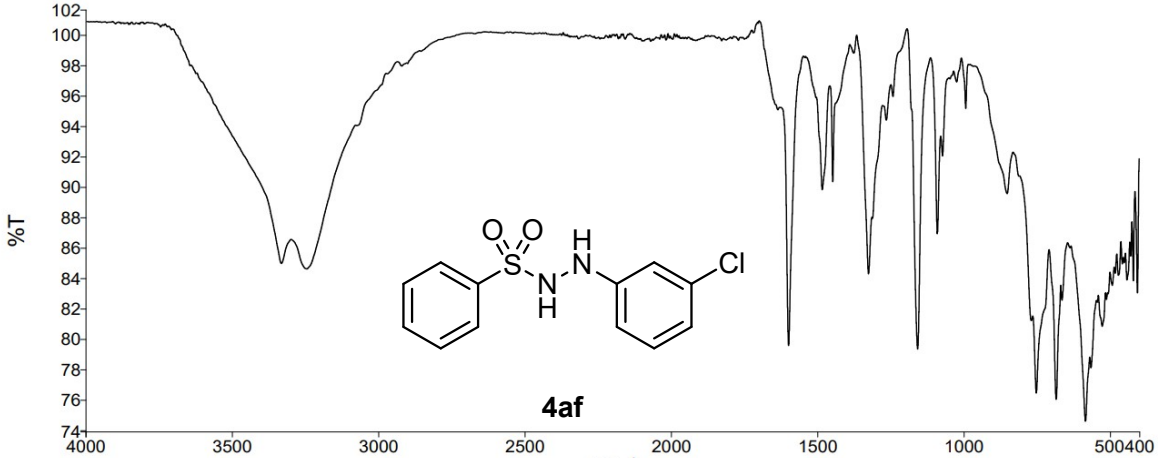
3.15. IR spectra of 4-methyl-N’,N’-diphenylbenzenesulfonylhydrazide (4be)



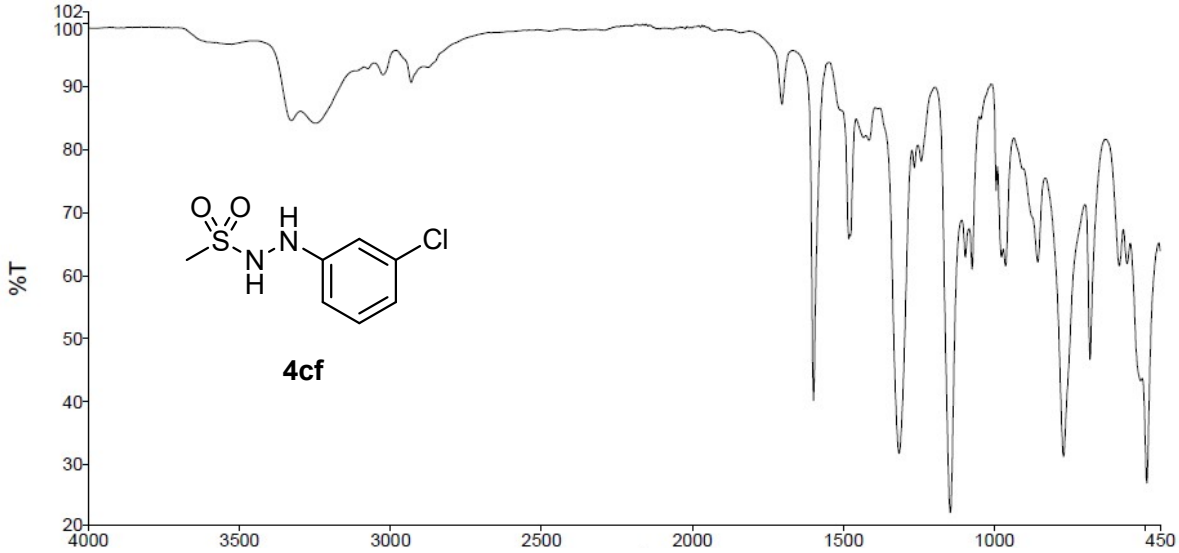
3.16. IR spectra of 1-methanesulfonyl-2-phenylhydrazine (4ce)



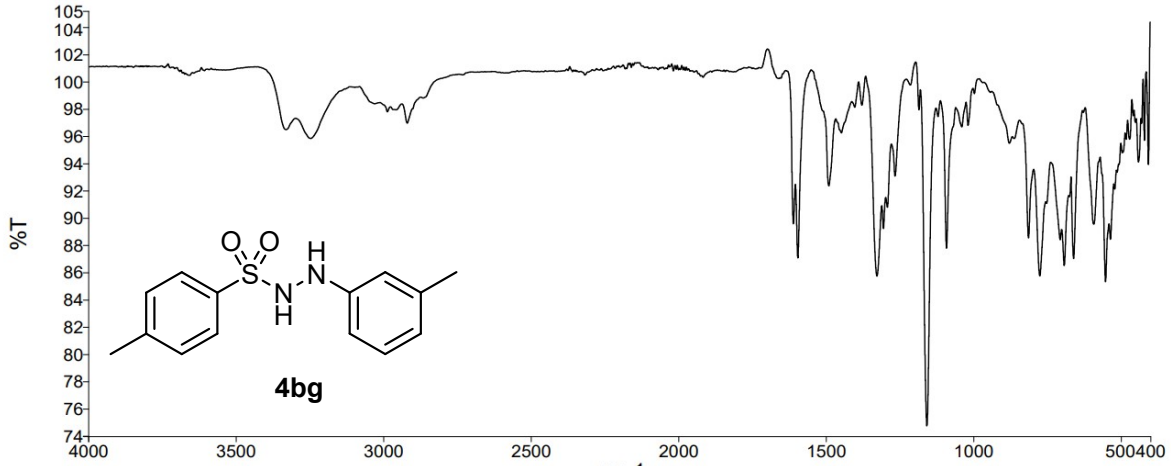
3.17. IR spectra of N'-(3-chlorophenyl)benzenesulfonohydrazide (4af)



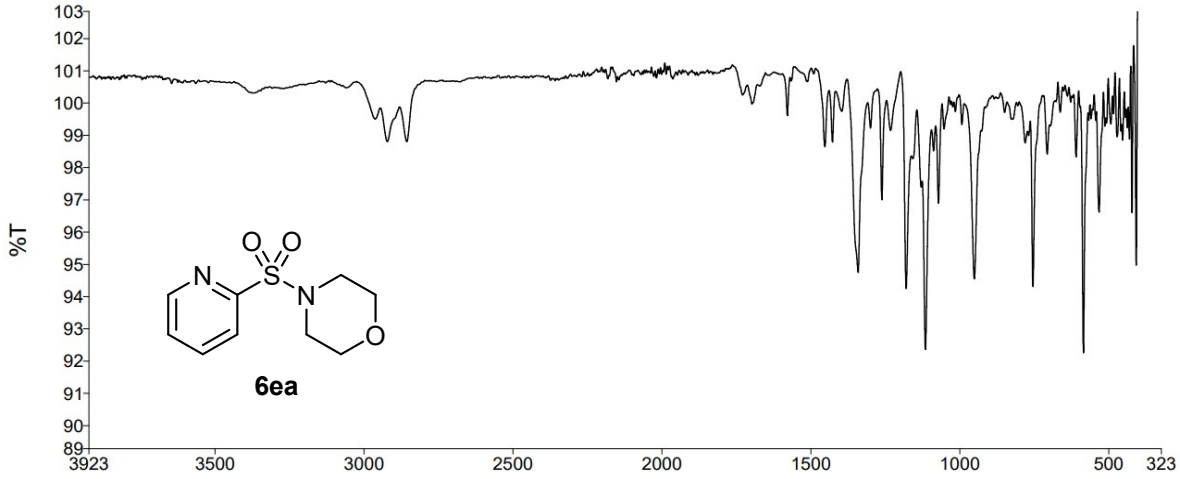
3.18. IR spectra of N'-(3-chlorophenyl)methanesulfonohydrazide (4cf)



3.19. IR spectra of 4-methyl-N'-(m-tolyl)benzenesulfonohydrazide (4bg)



3.20. IR spectra of 4-(pyridin-2-ylsulfonyl)morpholine (6ea)



4. Computational details

4.1. Energies and Cartesian Coordinates

R

SCF energy: −1165.01304164 Hartree

Free energy correction: 0.271778 Hartree

6	-0.717290000	4.584261000	3.058683000
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6	-0.986194000	2.581033000	1.698964000
6	-2.092590000	3.189557000	1.109788000
6	-2.537804000	4.466053000	1.454294000
6	-1.834036000	5.166856000	2.445528000
53	-3.152944000	2.116940000	-0.401229000
8	-4.275203000	4.281904000	-0.140081000
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6	0.921572000	-4.079148000	1.068103000
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8	2.613487000	-0.688389000	-0.957420000
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6	-1.889917000	1.334528000	-4.114181000
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1	-0.904867000	1.310178000	-2.170217000
1	-2.087806000	2.407585000	-4.053159000
1	-1.650395000	1.061421000	-5.157026000
1	-3.920568000	-1.184152000	-3.453918000
1	-2.707476000	-1.085340000	-4.772000000
1	-1.682882000	-2.281896000	-2.829988000
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TS

SCF energy: −1164.97718710 Hartree

Free energy correction: 0.271423 Hartree

Imaginary frequency: 319.9i

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6	-1.498107000	2.977268000	1.055529000
6	-2.498839000	3.344624000	1.963373000
6	-2.190405000	3.252101000	3.335648000
53	-1.829549000	2.998601000	-1.060534000
8	-4.090825000	4.076618000	0.327579000
6	-3.907009000	3.858907000	1.578595000

8	-4.720826000	4.018588000	2.544868000
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16	0.853760000	-0.316266000	-1.302729000
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6	-0.931212000	-0.606432000	0.848424000
6	-1.350963000	-1.163478000	2.064032000
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6	0.610356000	-2.610883000	2.087743000
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P

SCF energy: −1165.01273670 Hartree

Free energy correction: 0.273661 Hartree

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6	-1.689850000	2.892775000	1.849494000
6	-1.519666000	2.216178000	3.068944000
6	-0.193442000	1.914767000	3.450705000
53	-3.576998000	3.539118000	1.063615000
8	-3.842695000	1.849234000	3.629756000
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8	-0.616763000	1.470441000	-1.707556000
16	-0.029479000	0.142915000	-0.999874000
6	-0.998285000	-0.308598000	0.494315000
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6	-3.095680000	-0.369434000	1.641730000
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6	-0.327831000	-0.928211000	1.548050000
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1	0.433721000	-1.778455000	-2.316707000
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6	-0.332632000	-0.136723000	-5.806469000
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1	0.100207000	1.030256000	-4.018841000
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1	-0.219874000	-1.181763000	-6.143301000
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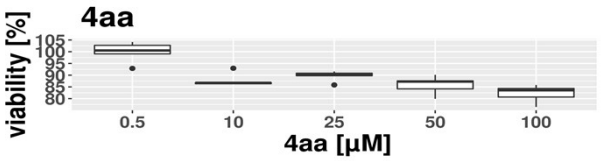
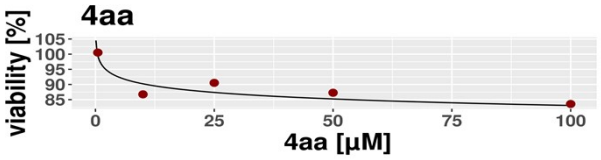
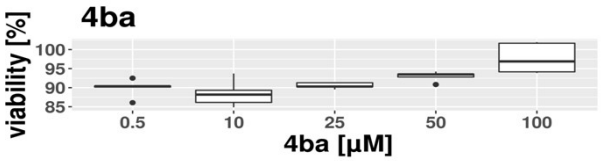
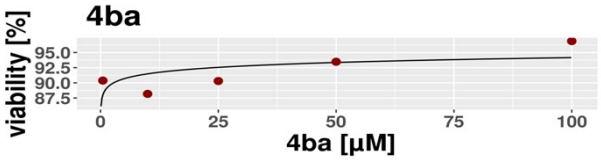
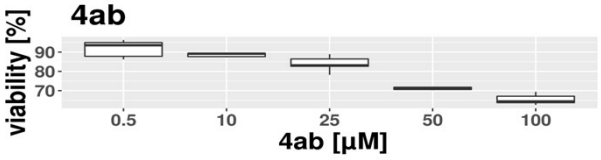
5. Cytotoxicity of Compounds

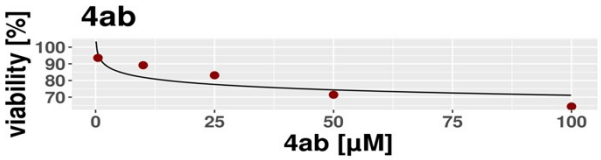


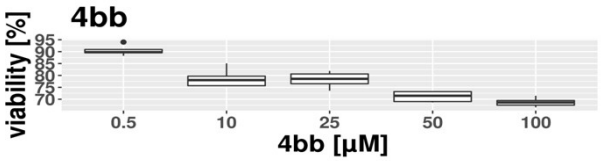


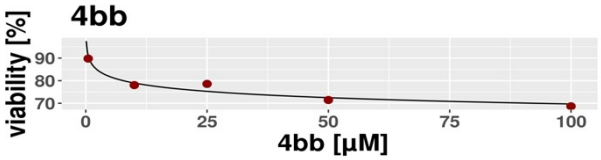


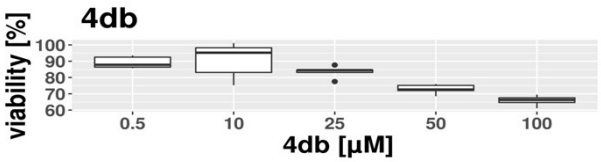


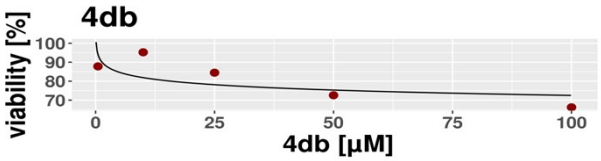


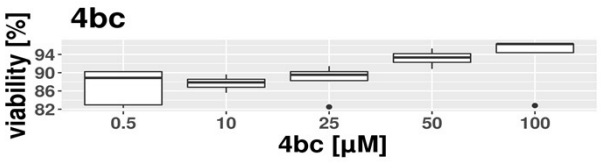


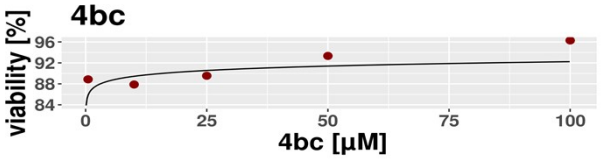


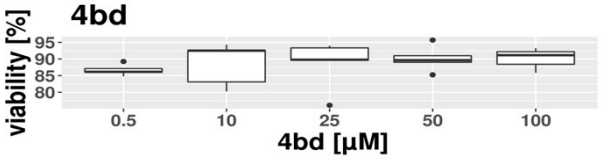


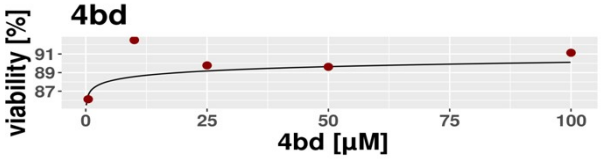


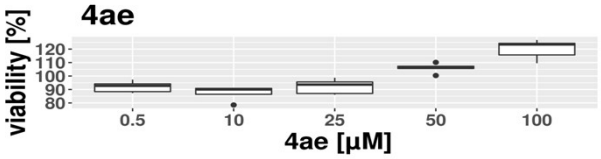


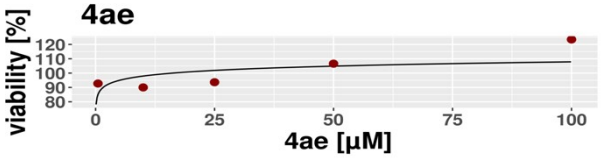

4bc
viability [%]
94
90
86
82
0.5
10
25
50
100
4bc [µM]

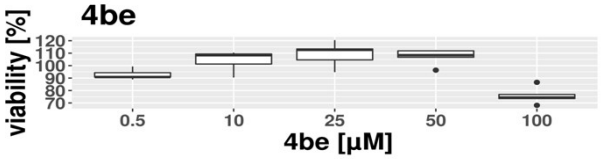

4bc
viability [%]
96
92
88
84
0
25
50
75
100
4bc [µM]

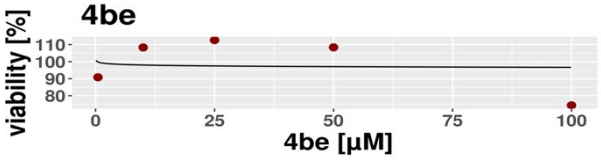

4bd
viability [%]
95
90
85
80
0.5
10
25
50
100
4bd [µM]

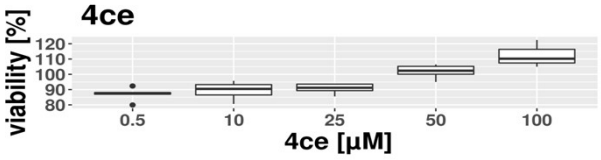

4bd
viability [%]
91
89
87
0
25
50
75
100
4bd [µM]

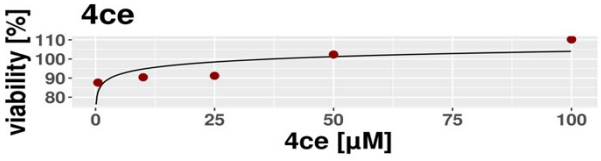

4ae
viability [%]
120
110
100
90
80
0.5
10
25
50
100
4ae [µM]

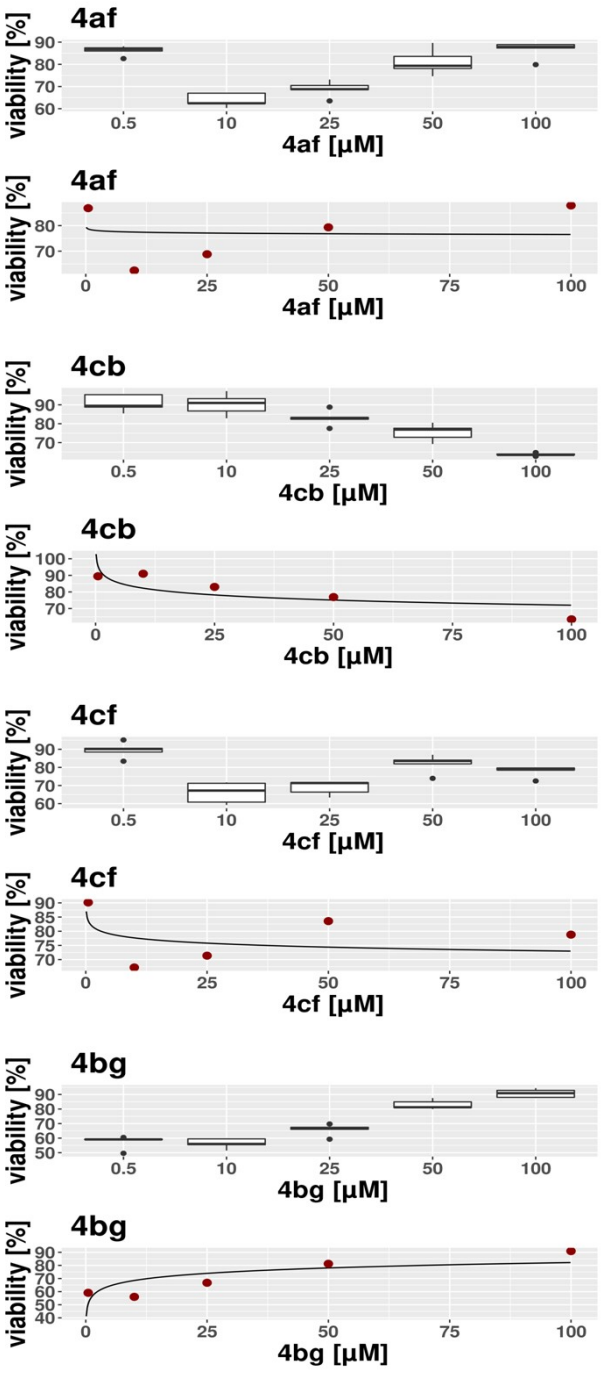

4ae
viability [%]
120
110
100
90
80
0
25
50
75
100
4ae [µM]


4be
viability [%]
120
110
100
90
80
70
0.5
10
25
50
100
4be [µM]


4be
viability [%]
110
100
90
80
0
25
50
75
100
4be [µM]


4ce
viability [%]
120
110
100
90
80
0.5
10
25
50
100
4ce [µM]


4ce
viability [%]
110
100
90
80
0
25
50
75
100
4ce [µM]



6. Availability of Data

The raw data for NMR analysis of the following target compounds are available through the Chemotion Repository (https://www.chemotion-repository.net/home/) under the collection DOI: https://dx.doi.org/10.14272/collection/CWG_2022-11-23.

1	https://dx.doi.org/10.14272/XSZMFYQLHGJKKD-UHFFFAOYSA-N.1
4aa	https://dx.doi.org/10.14272/DUAQQPBEBZYIHR-UHFFFAOYSA-N.1
4ba	https://dx.doi.org/10.14272/CGJMIHZEFDRIGG-UHFFFAOYSA-N.1
4ab	https://dx.doi.org/10.14272/MVFSSJKMQUDTLZ-UHFFFAOYSA-N.1
4bb	https://dx.doi.org/10.14272/JILSBUZMBFCCAQ-UHFFFAOYSA-N.1
4ac	https://dx.doi.org/10.14272/VXIQGBYMXSFXOT-UHFFFAOYSA-N.1
4bc	https://dx.doi.org/10.14272/LPASCALNKVOUAF-UHFFFAOYSA-N.1

4ad	https://dx.doi.org/10.14272/SZZHGCPDOORRHI-UHFFFAOYSA-N.1
4bd	https://dx.doi.org/10.14272/ZGKOFNKFGIVSDI-UHFFFAOYSA-N.1
4ae	https://dx.doi.org/10.14272/DGBYZIRNJBIXEI-UHFFFAOYSA-N.1
4be	https://dx.doi.org/10.14272/KXAFXMXACBYLEC-UHFFFAOYSA-N.1
4af	https://dx.doi.org/10.14272/NQGYJZMMGBFFFC-UHFFFAOYSA-N.1
4bg	https://dx.doi.org/10.14272/WKKLJJGGWZHEKG-UHFFFAOYSA-N.1
4cb	https://dx.doi.org/10.14272/CNJKVABMOKYTFU-UHFFFAOYSA-N.1
4db	https://dx.doi.org/10.14272/NZAOAPIMAXEJGE-UHFFFAOYSA-N.1
4ce	https://dx.doi.org/10.14272/RUXSYXRLQWWZKD-UHFFFAOYSA-N.1
4cf	https://dx.doi.org/10.14272/JJOSXMIVEFQUSL-UHFFFAOYSA-N.1
4eb	https://dx.doi.org/10.14272/BOGYYCZNODBDFD-UHFFFAOYSA-N.1